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Assessment of tire wear emission in a road tunnel, using benzothiazoles as tracer in tunnel wash water

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Water and Environmental Technology

Forord

Med denne oppgaven avslutter vi vårt femårige masterstudium i vann- og miljøteknikk, ved Fakultet for realfag og teknologi (REALTEK) på Norges miljø- og biovitenskapelige universitet (NMBU). Oppgaven tilsvarer 30 studiepoeng.

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Ås, May 14, 2019

Ninni Helene Bye & Jon Petter Johnsen

Abstract

This thesis was performed after recommendations by the Norwegian Public Road Administration in cooperation with the Norwegian institute for Water Research (NIVA), in order to develop a new methodology to quantify tire tread emission in the environment. The physical properties of the tire particle, makes it challenging to detect with existing visual detection methods for microplastics. Benzothiazoles (BTs), organic components in tire tread, has earlier been proposed as tracers for tire wear in road runoff, and was therefore investigated as a possible tracer for tire tread particles (TP) in this study.

Road tunnels are known hotspots for road pollutants due to the minor exposure to weather, and it was therefore expected high concentrations of tire wear accumulated in the tunnel. To maintain traffic safety in tunnels, the tunnels are washed frequently. The highly contaminated wash water contain tire wear particles and other road pollutants, and must be treated accordingly before discharge to nearest recipient. The Smestad tunnel, in Norway, is exposed to a relatively high annual average daily traffic (AADT) of 57 888 and has a treatment system for wash water. The tunnel was chosen as an ideal study location for this thesis.

During a tunnel wash, 22 water and sediment samples were collected from the tunnel wash water and analyzed for a selection of benzothiazoles; 2-hydroxybenzothiazole (OHBT), 2-mercaptobenzothiazole (MBT), 2-aminobenzothiazole (ABT), Benzothiazole (BT) and 2-methylthiobenzothiazole (MTBT). Through a comprehensive series of calculations and estimations based on the concentrations of BTs, the daily TP emission were estimated. For the measured concentrations of BTs, the daily TP emission in the tunnel was estimated to 2−2.8 kg TPs/day (by OHBT), 7.4−10.4 kg TPs/day (by ABT), 0.1−4.2 kg TPs/day (by BT), 0.6−3.4 kg TPs/day (by MTBT) and 2.8−4.0 kg TPs/day by the sum of BTs. The TP emissions estimated from measured concentrations in the wash water were compared to an estimated TP emission based on an equation by Vogelsang et al. 2019, using emission factors (TP

emission per vehicle kilometer (mg/vkm)) based on two different studies. The calculations which was based on the equation resulted in a TP emission of 3.3−4.1 kg TPs/day. The estimated TP emission, based on BTs from this study, seems to be comparable to other studies. The concentration of BTs used to estimate the TP emission, were detected in samples taken directly from the wash water runoff.

The samples throughout the treatment system gave mixed results, and gave little information on presence of tire wear particles (TWP). This is due to the rapid degradation and leaching of BTs. The tunnel wash water may be affecting the degradation of BTs, due to its basic characteristics and combination with soap. The samples collected in this study, are from one tunnel wash only and should be interpreted accordingly.

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Abbreviations

Acronym	**Explanation**
ABT	Aminobenzothiazole
BT	Benzothiazole
CRM	crumb rubber material
EU	European Union
HOC	Hydrophobic organic pollutants
MBT	Mercaptobenzothiazole
MTBT	Methylthiobenzothiazole
OHBT	Hydroxybenzothiazole
PAH	Polycyclic aromatic hydrocarbon
PCB	Polychlorinated biphenyl
PLC	Programmable logic controller
PMB	polymer modified bitumen
POP	Polychlorinatedbiphenyls
RP	Road particle
RAMP	Road dust-associated microplastic particles
RWP	Road wear particle
SBR	Styrene−butadiene rubber
SBS	Styrene−butadene-styrene
TP	Tire particle
TSS	Total suspended solids
TWP	Tire wear particle
UV	Ultraviolet
NIVA	Norwegian Institute for Water Research

1. Introduction

Traffic related contamination and polluted road runoff, has negative affect on the environment (Meland 2010a, Scher and Thièry 2005, Zimmermann et al. 2002). Heavy metals, PAHs and microplastics are some of the emissions from roads that is of great concern (Meland 2010a). Microplastics as pollutant debris are antrophogenic emissions, which has negative effects on terrestrial and aqatic living organisms (Horton et al. 2017) (Guzzetti et al. 2018), and the possibility that microplastics with its hazardous components and additives can be ingested by humans, makes this a subject of great importance for further investigation (Auta et al. 2017, Talsness et al. 2009, Van Cauwenberghe and Janssen 2014). Tire wear is a significant contributor to microplastic emission, but lack awareness in our society (Kole et al. 2017, Hartmann et al. 2019). Therefore it is recently designated as a "*stealthy source of microplastics in the environment*" (Kole et al. 2017).

The Norwegian Government stated in their National Transport Plan that the key to restrict microplastic debris from roads, is to detect and to increase focus on the sources (Norwegian Ministry of Transport and Communications 2017). It is crucial to address this potential threat in order to obtain knowledge on the subject, and to create new innovative methodologies to quantify and detect the extent of tire wear in the environment. Previously, there have been proposed that components in tires and additives can be suitable as tracers for urban runoff (Asheim 2018, Klöckner et al. 2019, Reddy and Quinn 1997, Spies et al. 1987,Pant and Harrison 2013), but it is challenging to find a suitable tracer to detect the presence of tire wear particles.

The thesis has focused on determining the amount of tire−related microplastic particles in a Norwegian road tunnel system with high average annual daily traffic (AADT). Tunnels are hotspots for road pollutants (Meland 2010a) and a washing sequence is important to maintain traffic safety (Statens Vegvesen 2014). The tunnel wash water is highly polluted (Meland2010a) and is led through the drainage system to a treatment system for wash water before discharge. The aim of the thesis is to assess the use of a suggested tire wear tracer, benzothiazoles (BTs), to quantify the emission of tire particles (TP) from vehicles in the road tunnel, and to determine the distribution and restraining of tire wear particles (TWP) in the treatment system, if possible. In order to predict the faith and distribution of TWPs in a road tunnel, there are numerous factors that needs to be considered for both the BTs and the TWPs itself. These factors are described in detail in this thesis.

To assess the behaviour and distribution of BTs, sediment and water samples were collected in each step of the treatment system. The samples were sent to a laboratory at The Norwegian Institute for Water Research (NIVA) for detection of five selected benzothiazoles; 2-hydroxybenzothiazole (OHBT), 2-mercaptobenzothiazole (MBT), 2-aminobenzothiazole (ABT), Benzothiazole (BT) and 2-methylthiobenzothiazole (MTBT). Analysis of the behaviour and tracer suitability of the benzothiazoles was studied in order to accomplish the following objectives:

Objective 1: Calculate tire wear emission in a tunnel based on the concentration of benzothiazoles in wash water runoff.

Objective 2: Assess the distribution of tire wear particles in a treatment system for tunnel wash water, based on concentrations of benzothiazoles.

To achieve these objectives, the following questions needs to be answered:

- Will degradation of the benzothiazoles interfere with our results?
- Does the pH in the tunnel wash water influence the degradation of benzothiazoles?
- Are the concentration of benzothiazoles in tunnel wash water suited for calculating tire particle emission?
- Will leakage of benzothiazoles from tire wear particles complicate our calculations?

2. Theory

2.1 Microplastics in the environment

Microplastics has no universal definition (Gigault et al. 2018), but are in various earlier studies defined as small particles of plastic, or rubber, in a study−specific size range from 1 nm to 5 mm (Kole et al. 2017, Talvitie et al. 2017, Hartmann et al. 2019). GESAMP 2015 included a particle size of nano−range in the microplastic definition, but in other studies the size range 1 nm−1 µm is defined as nanoplastics (Gigault et al. 2018). A more recent article by Hartmann et al. 2019 bases the size categorization of plastic on conventional unit of size, meaning SI prefixes for length, implying that microplastics are of size 1 µm−1000 µm.

Microplastics are classified as primary or secondary microplastics due to its original purpose when manufactured. Primary plastics are intentionally manufactured as small particles for industrial use, materials used in air−blasting technology, in self care products such as microbeads in exfoliants and in cosmetics. Secondary microplastics are particles derived from a larger plastic or rubber product, and develops through wear and tear of the product, weathering, biological and solar UV−degradation in the environment. Synthetic fibers in clothing, decomposition of plastic waste and wear of asphalt, road markings or vehicle tires are examples of secondary microplastics (GESAMP 2015, Kole et al. 2017).

The hazards due to plastic litter in the terrestrial and aquatic environment has been in the spotlight for decades (Gregory 2009). The recognizable damage of plastic debris are often associated to animals suffering from entanglement, suffocation or debilitation (Gregory 2009). In recent years, the smaller particles of plastics has gained attention, specifically in the marine environment (Carpenter and Smith 1972). Microplastics are present in the water column, in sediments and in biota worldwide (Van Cauwenberghe et al. 2015). The highest concentrations of microplastics are detected in industrialized and urbanized areas, and in harbours (OSPAR Commission 2017). Studies concerning marine environment have revealed the potential hazardous effects of microplastics in marine organisms (Guzzetti et al. 2018). Ingestion of microplastic particles can cause physical harm due to the particle itself which can lead to mechanical attachment on to the external surfaces, hindering mobility and clogging of the digestive tract in the animal/organism (Setälä et al. 2016). Animals/organisms can also suffer from chemical issues like inflammation, hepatic stress, decrease in growth, cancer, impaired reproductive activity, decreased immune response, and malformation (Auta et al. 2017). The microplastic particle can serve as a carrier of pathogens and potentially infect the consumer (Auta et al. 2017). Depending on composition and properties, the microplastic particles can absorb and accumulate persistent organic pollutants (POPs), also named hydrophobic organic pollutants (HOCs) (Cole et al. 2011), such as PAHs, PCBs, organochlorine pesticides (Wright and Kelly 2017), in addition to heavy metals and other toxins from the surrounding environment (Wright and Kelly 2017, GESAMP 2015).

Studies regarding human exposure to microplastics and its leached and adhered contaminants suggests numerous potentially hazardous effects in the human endocrine system, the reproductive system and the nervous system, even at low, environmental relevant concentrations (Talsness et al. 2009, Van Cauwenberghe and Janssen 2014). Humans are exposed to microplastics through inhalation and consumption. Dietary exposure happens through consumption of food with potentially bioaccumulated microplastics and its adherent pollutants through the food chain, or in drinking water (Wright and Kelly 2017, Pivokonsky et al. 2018). According to the OSPAR Commission 2017 the available data on microplastics, considering its occurrence, toxicity and fate when digested, are insufficient for a full risk assessment.

2.2 Road dust-associated microplastic particles (RAMP)

RAMP, and particularly tire wear particles (TWPs) which are kneaded with road pollutants (Hartmann et al. 2019, Adachi and Tainosho 2004), are potentially of severe concern (Chapter 2.4 Road dust and 2.6.2 Hazadous components). The microplastic particles, e.g. TWPs, can also leak inherent contaminants added during manufacturing, such as plastic additives, often termed plasticisers, which is harmful to marine biota (Cole et al. 2011, Talsness et al. 2009). As stated by Sundt et al. 2014, 53,6 % of the total amount of microplastic emission in Norway originates from tires and deserves attention accordingly. According to The Convention for the Protection of the Marine Environment of the North-East Atlantic (the "OSPAR Convention"), tire wear from vehicles is one of the main sources to microplastic in the marine environment in the OSPAR catchment, accompanied by land-based litter, each source with an estimated amount of 100 000 tonnes per year (OSPAR Commission 2017). Kole et al. 2017 estimated the total wear and tear of tires in the world to be 5 917 518 tonnes/year. That means with a population of 7 323 187 457 people in 2017, the amount of emitted tire tread debris per person equaled 0.81 kg/year (Kole et al. 2017). According to Vogelsang et al. 2019, 40-60% of the tire tread are different types of rubbers (e.g. microplastics), resulting in an yearly emission of minimum 0.5 kg of tire related microplastics per person in the world.

Currently there exists no legislation considering microplastic particles as contaminants in food (OSPAR Commission 2017), nor removal requirements of microplastics in wastewater or tunnel wash water before discharge to recipient (Vogelsang et al. 2019)(Chapter 2.6.3 Transport and 2.8.2 Discharge permits). According to Boulter 2005 and Zhang et al. 2018, there are no EU regulations designed to control the emissions of non- exhaust particle emissions, like TWPs. Due to the increased awareness regarding the negative impact from the transport sector (e.g. construction, manufacturing, maintenance of transport infrastructure) on the environment, the Norwegian Government aim to reduce the dispersion of plastics in order of protecting ecological and chemical water quality and biodiversity (Norwegian Ministry of Transport and Communications 2017).

2.3 Tire Particles

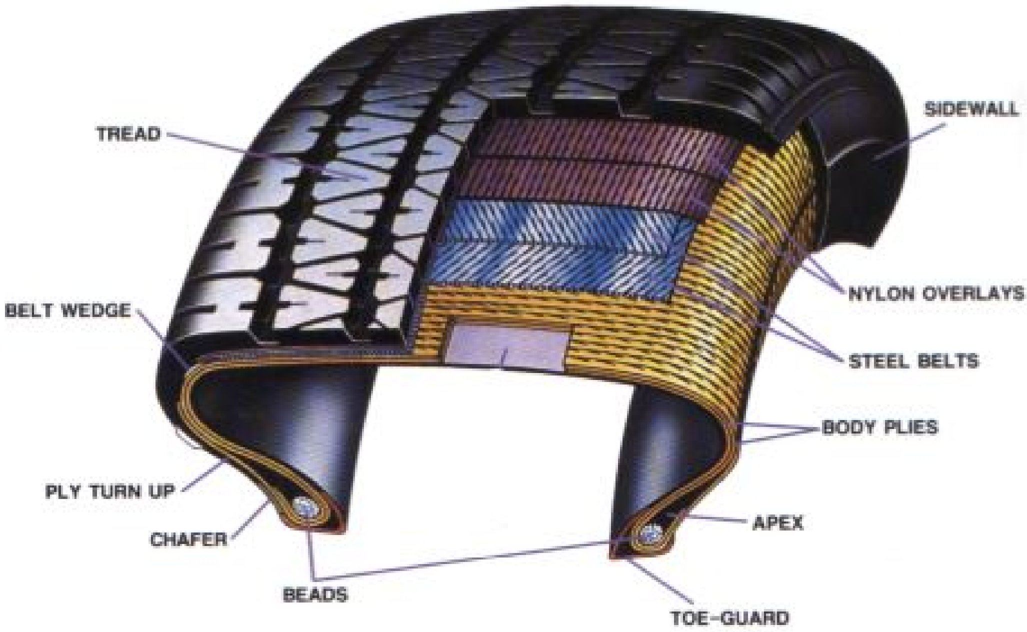

Figure 2.1: The composition of a passenger vehicle tire. The wear of tire tread causes microplastic emission as TWPs. Illustration copied from ChemRisk Inc. and DIK Inc. 2008

The composition of a tire depends on its application and the formulation varies between the different manufacturers and is often kept as a commercial secret (Boulter 2005). Generally, passenger car tires differ from tires of heavy- duty vehicles, and their composition consists of various combinations of synthetic and natural rubber, filler, accelerators, processing aids, reinforcement agents, adhesives, retarders and activators (Boulter 2005, Wagner et al. 2018). Because of its content, TPs has different characteristics than other microplastics, making the particles distribute differently in the environment and more challenging to detect visually (2.6.1 Physical characteristics). In the environment the tire particles mainly occur as tire wear particles (2.6 Tire wear particles), and these particles have different physical properties than TPs mechanically produced for scientific use or as artificial turf granules (Kreider et al. 2010).

Main elements in tire composition are organic zinc, which is found at the concentration 1.2 g/kg tire rubber (Boulter 2005). Other elements detected in significant amounts

Table 2.1: Composition of passenger tire tread. Copied and modified by Vogelsang et al. 2019

Component	**Content**	**Ingredients**	**Typical content**	**Plastic?**
Rubber polymer	40−60%	Natural rubber co-polymers	16−24%	Yes
		Styrene- butadiene rubber (SBR)	12−18%	Yes
		Poly- butadiene rubber (PBR)	8−12%	Yes
		Other rubbers	4−6%	Yes
Reinforcing agent (filler)	20−35%	Carbon black (to improve hardness and wear resistance)		No
		Carbon black recently partially substituted with silica incorporated with a silane coupling agent, carbon- silica dual- phase filler (CSDP) and/or "nano structure" carbon blacks (to increase rolling resistance without compromising strength and longevity).		No
Process oil/ extender oils	15−20%	High aromatic oils rich in poly aromatic hydrocarbons (PAHs)		No
		The use of mild extract solvates (MES) and treated destillate aromatic extracts (TBAE) is increasing.		No
Vulcanization agents	1%	S, Se, Te, thiazoles, organic peroxides, nitro- compounds (to improve the durability of tire rubber)		No
Vulcanization activators	2.5%	Zinc oxide	1.5%	No
		Stearic acid	1%	No
Vulcanization accelerators	0.5%	Lead, magnesium, zinc, sulphur compounds (sulphonamide or thiazoles) and calcium oxides.		No
Vulcanization retardants		Terpene- resin acid blends		
Protective agents	1%	Preservatives (halogenated cyanoalkanes), anti-oxidants (amines, phenols), anti-ozonants (diamines and waxes) and desiccants (calcium oxides)		No
Processing aids	<1%	Peptisers		
		Plasticizers (synthetic organic oils and resins) (to provide elasticity and stickiness to the tire)		
		Softeners		

are calcium, aluminum, barium, potassium, sodium, iron, copper and titanium though neither as prominent amounts as zinc (Boulter 2005). A truck tire typically contains 80% natural rubber, whereas a tire of a passenger car contains approximately 15% natural rubber (Camatini et al. 2001). A high content of natural rubber in tires increases its tear strength and resistance to heat. The elastic and flexible raw natural rubber can through the vulcanization process with sulfur, or hardening with carbon black filler, achieve crystallization-induced strength and toughness, due to its polymer network (Baumann and Ismeier 1998, as referenced in Wagner et al. 2018). This makes natural rubber a strong material preferred in heavy duty vehicles like trucks and buses (Wagner et al. 2018). A mix of styrene-butadiene rubber (SBR) and polybutadiene rubber (PBR) are dominant in tires of passenger cars (Vogelsang et al. 2019). Non- studded tires contains a larger amount of PBR than summer tires hence its necessary with a softer texture that improves grip (Vogelsang et al. 2019). Accelerators are added to increase the efficiency of the vulcanization process (hardening of rubber)(Boulter 2005, Akiba and Hashim 1997). Benzothiazoles are degradation products of this process and are in various studies proposed as markers for tire debris (2.9 Benzothiazoles) (Boulter 2005, Reddy and Quinn 1997). Tire tread is a well mixed material, and the composition is the same in the finer and the coarse particle fractions (Rogge et al. 1993).

2.4 Road dust

Road dust is a heterogeneous mix of particulate matter from a variety of contributing sources such as vehicle related exhaust and non-exhaust particles, particles from abrasion of the paved road, soil and biogenic materials from surrounding environment (Rogge et al. 1993). The road dust contains accumulated toxic compounds which can be distributed in the atmosphere by wind or vehicle induced turbulence, or to the hydrosphere by road runoff (Subchapter 2.6.3)(Rogge et al. 1993) or by highly contaminated wastewater (Chapter 2.8.2). Some examples of road associated main pollutants are PAHs, Oils and heavy metals like Cu, Zn, Pb, Ni, Cr and Cd (Meland 2010a).

The sources of road dust-associated microplastic particles (RAMP), are presented in Figure 2.2 to illustrate the connections between the terminologies. A pure tire tread particle (TP) are derived directly from the tire tread. The tire wear particle (TWP) are a tire particle generated by driving and is found in the environment (Chapter 2.6), often mixed with other road particles (RPs). The RPs are all kinds of particles located on the road (e.g. minerals). A particle generated by wear of the road pavement is termed a road wear particle (RWP) and it is distinguished between the RWPs originating from the road marking (RWP_{RM}) containing thermoplastic elastomers, and the (RWP_{PMB}) containing polymer from the polymer modified bitumen (PMB) in the wear layer of the road.

$\text{RAMP} = \text{TWP} + \text{RWP}_{RM} + \text{RWP}_{PMB}$

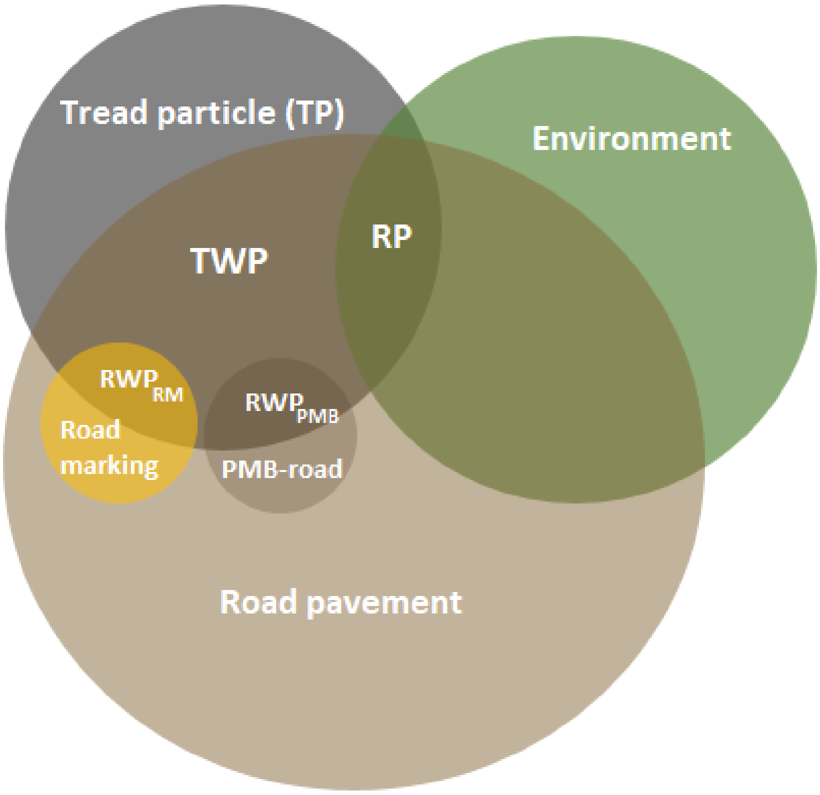

Figure 2.2: Illustration of contributors to road dust associated microplastics (RAMP). Modified and copied from Vogelsang et al. 2019

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According to Amato et al. 2014, 96% of the general road dust mass accumulation (i.e. RPs) was identified produced from three main sources: Carbonaceous sources, mainly related to abrasion of tires (i.e. TWPs) with motor exhaust and brake wear as possible contributors, RWPs/minerals and break wear. The source distributions are illustrated in Figure 2.3.

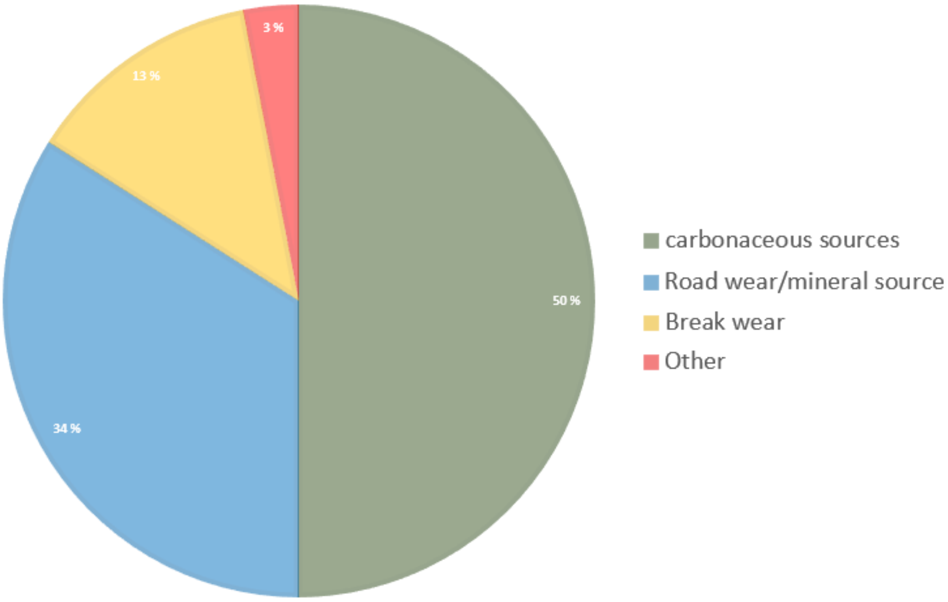

Figure 2.3: Contributing sources of road dust. Numbers copied from Amato et al. 2014

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The road dust contains microplastics where the main contributors are believed to be the rubber in tire treads, followed by thermoplastic elastomers in road marking and polymers added to strengthen the bitumen in asphalt (Vogelsang et al. 2019). Vogelsang et al. 2019 presented the following amounts of RAMP contribution:

- TWP: 4 300−5 700 tonnes of microplastics/year.
- RWP$_{RM}$: 28 tonnes of SBS/year
- RWP$_{PMB}$: 90−180 tonnes thermoplastic elastomers/year

2.5 Road wear particles

RWPs are developed during wear of the road surface, mainly due to vehicle traffic (Vogelsang et al. 2019). The bitumen are added to the asphalt to increase its resistance against road traffic, mainly due to the use of studded tires, and climate (Jørgensen et al. 2016). A variety of polymers are added to the bitumen (PMB, short for polymer modified bitumen) to ensure good properties at low temperatures, such as elasticity and flexibility (Jørgensen et al. 2016). These properties can prevent the asphalt from cracking, in addition to increase its stability during higher temperatures, making it stiffer and prevent rutting (Jørgensen et al. 2016, Sengoz and Isikyakar 2008). Some popular polymer additions are polyethylene (PE), polypropylene (PP) and (styrene-butadiene styrene (SBS) (Zhu et al. 2014). The thermoplastic elastomers used in road marking paints are Styrene-isoprene-styrene (SIS), Ethylene vinyl acetate (EVA), Polyamide (PA) and Acrylate polymers Sundt et al. 2014. There exists asphalt containing crumb rubber material (CRM), often from old tires, that may leach contaminants in the environment Reddy and Quinn 1997. CRM asphalt have been used in Canada and USA for decades, and in the later years in some European countries (Presti 2013, *Norsk dekkretur*).

2.6 Tire wear particles

The TWP is a byproduct caused by interaction between tire tread and carriageway. Micro vibrations from friction and stiction will wear the tire over time (Fukahori and Yamazaki 1995) and generate TWPs through rolling shear (Rogge et al. 1993, Sommer et al. 2018). Presence of studs will generate 16% less TWPs compared to regular summer tires (Snilsberg 2008). A TWP does not only consist of pure TP, but will aggregate to other traffic related sources (Chapter 2.4) (break wear, pavement, road furniture etc.) and can possibly be of great threat to surrounding environment (Adachi and Tainosho 2004). The TWP can be difficult to detect, due to its interaction with other particulate matter causes changes in composition and size. There are numerous factors contributing the quantity of tire wear and deposition of TWP (listed in Table 2.2).

Table 2.2: Factors contributing to the wear of tires. Copied from Boulter 2005.

Tire charachteristics	**Road surface characteristics**	**Vehicle characteristics**	**Vehicle operation**
Size (radius/width)	Material (bitumen/concrete)	Weight	Speed
Tread depth	Texture pattern and wave length	Distribution of load	Linear acceleration
Presence of studs	Surface dressing (road salt/sprinkle sand)	Location of driving wheels	Radial acceleration
Construction	Wetness	Engine power	Frequency of breaking/cornering
Pressure and temperature	Condition	Electronic breaking systems	Extend of breaking and cornering
Contact patch area		Suspension type	
Chemical composition		State of maintenance	
Accumulated mileage			
Set up			

2.6.1 Physical characteristics of TWPs

The morphology of TWPs are generated by rolling shear of tire tread against the road surface, giving the particles their characteristic elongated, or “sausage” like shape (Figure 2.4) (Vogelsang et al. 2019, Adachi and Tainosho 2004, Kreider et al. 2010). The TP consists of carbon black (>20% dw) that makes the TWP hard to detect visually with its dark color and its ability to blend in with other dark particles in the road dust (Kole et al. 2017). The high mineral content (>50% dw) in TWPs (Unice et al. 2013, Kreider et al. 2010) is caused by the melting process through friction forces acting between tire and carriageway, giving the TWPs a higher density (specific gravity) relative to water. Densities of TWPs will vary from 1.7–2.1 g/cm^3 (Vogelsang et al. 2019) compared to TPs with an average density of 1.15 g/cm^3 (Heitzman 1992). The size distribution of TWPs is crucial when predicting TWPs fate in the environment and for which treatment to apply. Kreider et al. 2010 found that the majority of TWPs was found around 75 µm. This corresponds well with earlier studies concerning TWP (Smolders and Degryse 2002). Given this data, a TWP with a diameter of 75 µm and a density of 2 g/cm^3 will approximately settle with a speed of 8.4 m/h, using Stoke’s Law for freshwater.

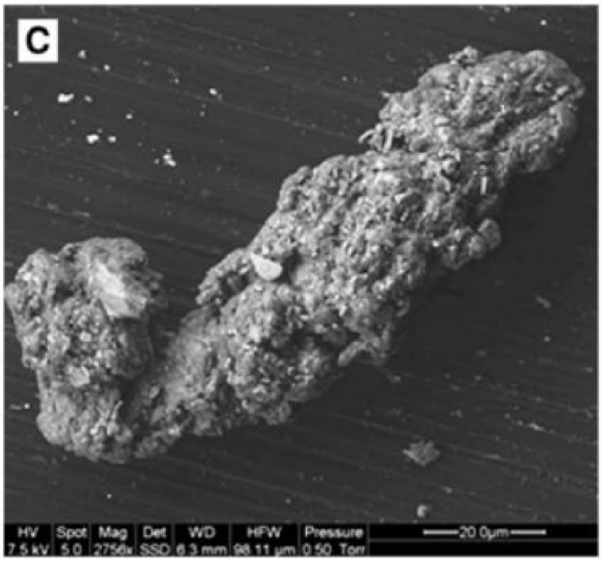

Figure 2.4: A tire wear particle (TWP) kneaded with minerals from carriageway. Copied by Kreider et al. 2010

2.6.2 Hazardous components in TWPs

The TWP and TP has shown ability to adsorb and accumulate other toxins as mentioned in "microplastics in environment" (Chapter 2.1). Tire abrasion is implicated as a source of heavy metals and other chemical contaminants entering the environment. There have been several studies concerning TP and its effects on biota (Gualtieri et al. 2005, Day et al. 1993, Ahlbom and Duus 2003). Various environmental pollutants used in tire manufacturing (e.g. benzothiazoles, Chapter 2.9) may leach out of TWPs (Gualtieri et al. 2005). In fact, there are reports showing that leakage of pollutants from tires exposed to water are size dependent, indicating that smaller particles with a greater exposed surface will leach more heavy metals and pollutants into the water phase (Gualtieri et al. 2005).

Zinc is highly present in TP (1-2% by weight)(Rhodes et al. 2012, Councell et al. 2004) and has been suggested as a marker for tire wear (Fauser et al. 1999, Klöckner et al. 2019). Zn is also present in brake wear and other traffic related sources, which should make it unsuitable as a marker for tire wear (Grigoratos and Martini 2015). Despite this, German scientists recently indicated that Zn may be a good indicator when sampling from high traffic-influenced environments (Klöckner et al. 2019). Heavy metals are common pollutants in urban environments, where Zn is one of the biggest threats with its large quantity, toxicity and its mobility in water (Wik et al. 2008, Zheng et al. 2010, Durand et al. 2004, Councell et al. 2004, Davis et al. 2001). It is shown in a study by Aasum 2013 that the mobility of heavy metals, including Zn, will increase even more with the use of soap during a tunnel wash (2.8.1 Tunnel maintenance). The toxicity and bioavailability of zinc and other heavy metals is pH dependent (Gualtieri et al. 2005), with negative effects on various aquatic organisms like worms, fleas and fish (Schubauer-Berigan et al. 1993).

Kreider et al. 2010 collected RP, TWP and TP from asphalt-based roads in France and Germany, which has approximately the same characteristics as in rest of Europe. They found that RP and TWP had 95% more PAHs attached to the particles than TP. This corresponds to other studies that concludes that tires do not contribute much to the total PAH in road dust and that the PAHs are significantly related to other sources (Macıas-Zamora et al. 2002, Zakaria et al. 2002, Ahlbom and Duus 2003). This indicates that TWPs are possible carriers of pollutants. In order to provide protection to human health and the environment, the use of extender oils containing PAHs in tire manufacturing were banned in 2010 (Wik and Dave 2009).

2.6.3 Transport of TWP to environment

Though tire wear is constantly generated from traffic, the TWP are not accumulated at the same rate, due to its mobility in water (Kumata et al. 2002). There are found, and expected, large amounts of TWPs and its tracer elements in the road verge and soils several kilometers away from the original source, caused by transportation of particles and as suspended solids (SS) in air and water (Spies et al. 1987, Kole et al. 2017). Wind and water are two contributing transportation factors of particle dispersion in the environment (Figure 2.4), especially the latter together with snow melt (Kumata et al. 2002). Snow is able to accumulate both airborne pollutants under precipitation and at ground level (Stationary snow) (Van Noort and Wondergem 1985). The highly contaminated snow will melt during the spring and transport particles (Sansalone and Glenn 2002).

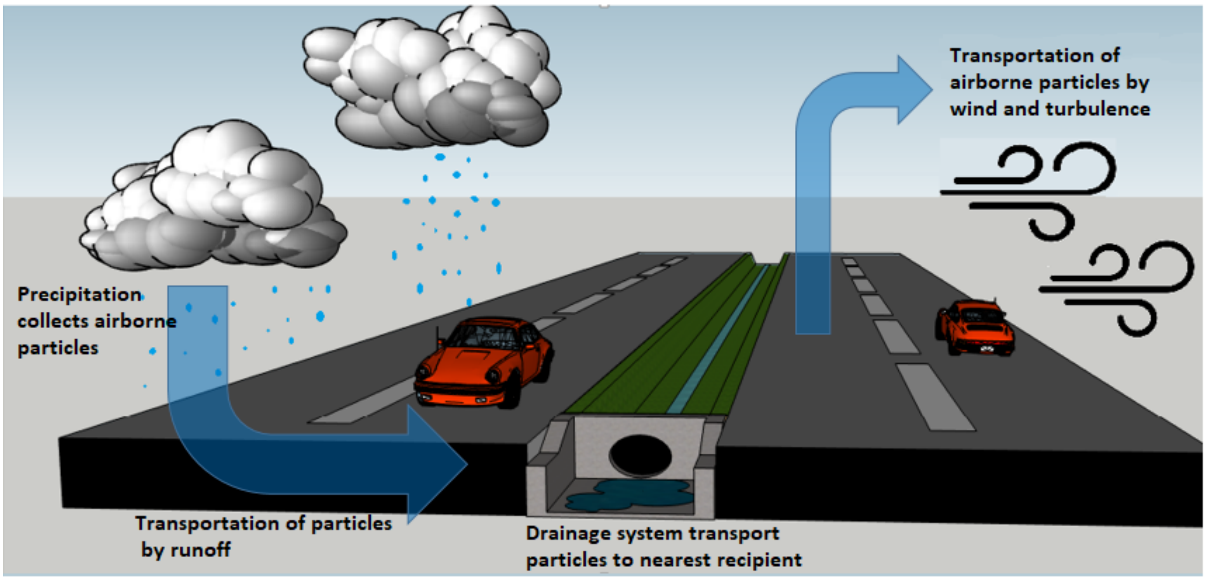

Figure 2.5: Conceptual drawing of the main transportation paths of TWP.

As mentioned in Chapter 2.6.1, the majority of TWPs is around 75 µm, meaning they will not be airborne for significant amount of time, so transportation by wind is a minor factor compared to water (Cadle and Williams 1978). The finer particles will also accumulate in the road structure where the wind has limited impact. Urban runoff will have a higher concentration of TWPs due to the lack of infiltration and less water is needed to transport particles over dense surfaces (Vogelsang et al. 2019). There are often municipal drainage systems in urban areas for transporting stormwater to the nearest recipient. If the recipient can not withstand the loading, treatment is required (2.8.2 Discharge permits). All discharges to recipients need to take the vulnerability of the recipient into account according to Water Framework Directive (WFD) in the Norwegian Water Regulation (Vannforskriften 2007), however there are no emission standards for TWPs, or non-exhaust emission in general.

2.7 Estimation of road associated tire emission in the environment

There are different methods for estimating TP emission. One method is an equation developed by Vogelsang et al. 2019 which requires the length of the road stretch investigated, annual average daily traffic (AADT) at the specific road stretch, and TP emission per vehicle kilometer (emission factor). The emission factor depends on what contributes to the tire wear process, such as driving style and weight of vehicle, and various studies show large variations in these wear estimations. The emission factors presented in Table 2.3 are based on a study in the Netherlands by Klein et al. 2017 and are adjusted to fit the information available at the Norwegian Road Administration online map service (Statens Vegvesen 2019) with data relating to AADT. Heavy duty vehicles are a category that includes buses, lorries and trucks, and the estimated tread is an average of the three. Urban driving are characterized by more frequent acceleration, braking and oscillation, and this will cause a higher rate of tire wear (2.6 Tire wear particles). Another study of emission factor, by Boulter 2005, estimated that for "normal" driving conditions a passenger car would release 100 mg per vehicle- kilometre (mg/vkm) of tire tread, while for heavy duty vehicles, an emission of 1000 mg/vkm. The latter estimation is based on a large span in reporting numbers from various studies, ranging from 136 mg/vkm (Legret and Pagotto 1999) to 1403 mg/vkm (SENCO 1999 referenced in Boulter 2005). The equation are presented in material and method, as Equation 3.1.

Table 2.3: TP emission based on vehicle and driving category. Collected data by Klein et al. 2017.

Vehicle (*i*)	**Urban driving (*j*) mg/vkm**	**Rural driving (*j*) mg/vkm**	**Highway driving (*j*) mg/vkm**
Passenger car	132	85	104
Heavy duty vehicle	641	412	504

The first method can be used to estimate the TP emission of a particular road stretch of interest, or if all the parameters are available, estimation of annual TP emission are possible. A second method for estimating the annual emission of tire wear is by weight loss of worn out and discarded tires. Sundt et al. 2014 estimated 9 600 tonnes tire tread/year in Norway (2.4 Road dust) based on this method. These methods for calculating TP emission, would require several road−specific parameters and will not consider transportation or distribution of TWPs in the environment. Therefore, it is proposed tracer elements to detect physical presence of TWPs (Pant and Harrison 2013, Asheim 2018, Wagner et al. 2018).

Wagner et al. 2018 developed an analytical strategy to determine the suitability of TWP tracers present in either rubber (SBR or natural rubber) as a major constituent of tires, or in additive from tire materials. They should ideally fulfill certain criteria as possible tracers, which is further explained underneath as a checklist:

- Be present in all tire materials in comparable portion, largely independent from manufacturer or manufacturing process.
- Not leach easily from tire particles into the surrounding environment
- Not be easily transformed while the tire particles reside in the environment (air, water, soil)
- Be sufficiently specific for tires, namely not present in relevant concentration in other traffic related particulate matter, such as brake dust, roadway particles or surface runoff.
- Have a concentration in tire material significantly higher than in the particles forming the sample matrix (soil, sediment, road dust, suspended matter, aerosol).
- Be analytically accessible by methods of high precision, accuracy, and sensitivity at reasonable analytical effort.

2.8 Road tunnels

Tunnels are hotspots for pollution and traffic related contamination, due to their accumulating properties. The low impact of wind and weather regarding transportation of particles, makes tunnels convenient sampling sites for road dust and traffic related pollution.

2.8.1 Tunnel maintenance

There are over 1200 tunnels in Norway with a combined tunnel stretch of >1400 km (Statens Vegvesen 2019), and they all have requirements to maintain traffic safety and give the drivers a positive experience throughout the tunnel (Statens Vegvesen 2014). With a high humidity and with concentrations of accumulated dust and gasses, the tunnels are designated as "highly aggressive environments" (Foslie and Chiodini 2010).

Figure 2.6: Tunnel wash. Photo: Jon Petter Johnsen.

Combined with high AADT, the tunnel wash water may exceed the discharge permits and the environmental quality standards (Meland et al. 2010b, Meland and E. Rødland 2018). To obtain the requirements set by Norwegian Public Roads Administration, the tunnels is swiped for dust and gravel. Subsequently the tunnels' sealing, walls, carriageway and technical gear are coated with soap and washed with high−pressure. Washing intervals like this depends on the traffic loading, or annual average daily traffic (AADT) (Table 2.4). The washing processes is distinguished between full- and half wash in addition of cleaning of technical gear. A full wash includes cleaning ventilators, sealing and emptying gully pots. A typical tunnel wash will start up with a mobile sweeper that removes surface grit and dust from pavement and carriageway to prevent clogging the stormwater pipes during a washing sequence.

Table 2.4: Wash frequency determined by AADT developed by the Norwegian Public Roads Administration (Statens Vegvesen 2014)

AADT per tunnel bound	**Full wash**	**In addition: half wash**	**In addition: Technical gear**
0-300	each 5. year	—	each year, in years without full wash
301-4 000	1 per year	—	1 per year
4 001-8 000	1 per year	1 per year	2 per year
8 001-12 000	1 per year	2 per year	3 per year
12 001-15 000	2 per year	3 per year	5 per year
15 001<	2 per year	4 per year	6 per year

2.8.2 Treatment of tunnel wash water

Meland 2010 referred to the tunnel wash water as a cocktail of contaminants and several other studies show that wastewater from tunnel wash, combined with soap, is potentially acute toxic to aquatic organisms (Meland 2010a). Studies show that soap may increase the pH in tunnel wash water, respectively pH 7.7−8.8 by Meland et al. 2011 and pH 7−10 by Aasum 2013, dependent on soap concentrations. Norwegian Public Roads Administration did a mapping of 74 tunnels in region east in 2013 (Torp 2013). They found that 58 (78%) of the tunnels did not treat the tunnel wash water before emission (Torp 2013). There are no specific guidelines to determine which tunnel pollute more than the other, but E. S. Rødland and Helgadottir 2018 made a useful tool to prioritize tunnels in Norway with SS, AADT and tunnel length as parameters (Figure 2.7). They used a five-step scale, where 5 is low priority and 1 is high priority.

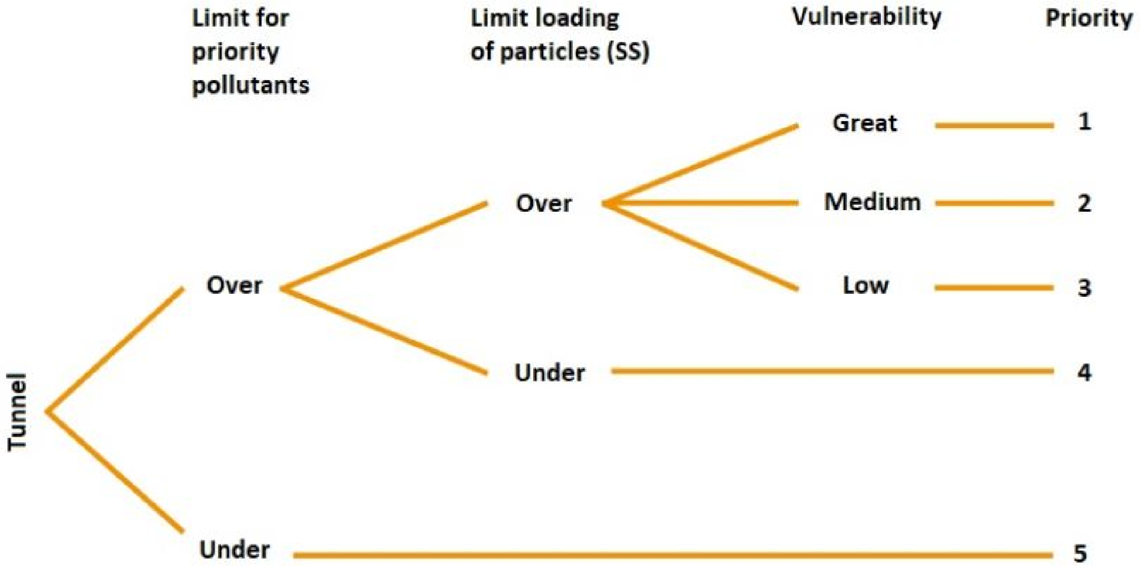

Figure 2.7: Schematic presentation of the priority tool. (Modified and borrowed from (E. S. Rødland and Helgadottir 2018))

Norwegian Public Roads Administration has set the treatment limits for regular road runoff according to the AADT and the recipients vulnerability. For tunnel wash waters however, it is required to apply for a concession to discharge the wash water to recipient. If the concession demands a treatment process, the treatment must minimum include sedimentation of particles and degradation of soap in closed basins, and also withhold oil, using a oil separator. The treatment facilities should also be able to withhold equivalent volume of water used during a full wash (Statens Vegvesen 2016). Therefore the treatment of tunnel wash water is an important step to reduce the pollutant loading in the environment, and it is therefore recommended that treatment systems should be mandatory when constructing new tunnels (Meland 2010a, Billiard et al. 1999, Byman 2012).

Gully pots

Common practice in tunnels are storm water pipes with catch basins, or “gully pots” as illustrated in Figure 2.8. Even though gully pots can restrain particulate−bound contaminants (Lindholm 2015), they are not considered a treatment process. Gully pots are mainly designed for reducing the risk of clogging the pipes and protecting downstream drainage. Lindholm 2015 stated that if gully pots do not exceed 50% filling level of grit, they can receive a maximum discharge of 20−25 L/s and still restrain 50% of heavy metals and withhold bigger particles like grit and sand (Lager et al. 1977, Lindholm 2015). Contaminants and heavy metals will be found both as dissolved and particulate−bound in the water phase (Sansalone and Buchberger 1997, Paruch and Roseth 2008). Dissolved material will not be restrained in gully pots designed for grit removal (Lindholm 2015,Lindholm 2015). Sprinkle sand is one of the contributing media that fill the gully pots over time, and is frequently used on Norwegian roads during the winter season. It is important that these gully pots are emptied regularly to obtain their potential (Lindholm 2015).

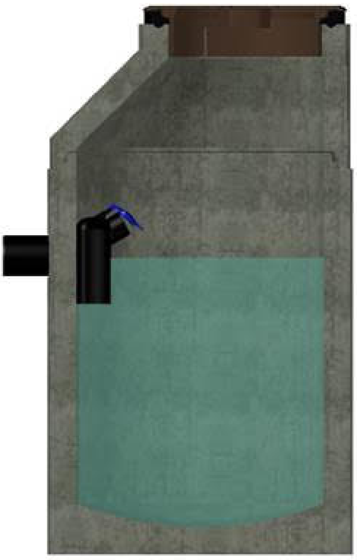

Figure 2.8: Cross section of a typical gully pot used on Norwegian roads with submerged outlet to remove oil and floatable contaminants. Copied and illustrated by BASAL AS

Sedimentation basins

There are various methods for treating tunnel wash water, but sedimentation ponds/basins are the most common process in Norway today (Meland 2010a, Aasum 2013). Efficiency of sedimentation basins are dependent on fraction size and density of the particles that is settling (Stoke's Law). Paruch and Roseth 2008 found that most contaminants in tunnel wash water were particulate−bound and could easily settle (Paruch and Roseth 2008). Sedimentation basins will not rinse dissolved material, (Pettersson 1999), but studies estimates that some dissolved components may adsorb to particles and therefor be expected to settle (Chapter 2.9.1 Soil/sediment adsorption coefficient). Degradation of toxic soap components used during tunnel wash is important and can be achieved by sufficient retention time in the sedimentation basins (minimum 2 weeks) under aerobic conditions (Åstebøl 2014). It is also shown that soap, and corresponding pH, will reduce the sedimentation efficiency of heavy metals (Aasum 2013, Brownlee et al. 1992). The retention time is also affected by the settling speed of particles to fulfill the treatment requirements (Åstebøl 2014). Sedimentation ponds (open ponds) are no longer recommended since the settling water are highly contaminated and can cause death to amphibians and other aquatic living organisms (Meland et al. 2010b).

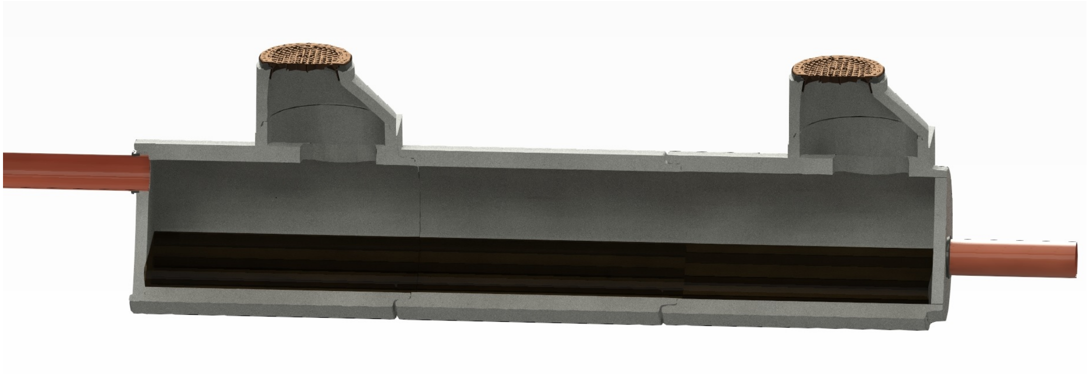

Figure 2.9: Cross section of a typical cylindrical sedimentation basin. Copied and illustrated by BASAL AS.

Infiltration

Nature-based treatment of tunnel wash water, like infiltration trenches, swales or constructed wetlands, are also functional treatment processes, often combined with pre-sedimentation. Infiltration is commonly used in different kinds of treatment and can restrain dissolved contaminants as well as plants, in the top layer (constructed wetlands), can adsorb and accumulate metals and decrease the concentration of pathogens (Kadlec et al. 2017). Uptake of pathogens and dissolved matter will happen in the unsaturated zone (aerobic condition) (Åstebøl 2014). Constructed wetlands and swales can also be of aesthetic contribution to our society.

2.9 Benzothiazoles as tracers for tire wear

Benzothiazoles and its derivatives are are high production volume chemicals utilized in various applications and are manufactured worldwide (De Wever and Verachtert 1997). Benzothiazoles (BTs) are organic heterocyclic compounds consisting of a 1,3−thiazole ring fused to a benzene ring (HMDB, 2018). Studies show that humans are exposed to benzothiazoles in many various environments. BTs have been detected in indoor dust Wang et al. 2013, tap water (Wang et al. 2016), antifreeze Wenderoth et al. 2004, textiles and tires (Avagyan et al. 2013). Since benzothiazoles are components in tires, it has been suggested as indicators of urban runoff (Reddy and Quinn 1997, Kumata et al. 2002, Spies et al. 1987). The investigated benzothiazoles are presented with characteristics relevant for this thesis in Table 2.5. The following subchapters presents environmental behavior of BTs, which is important when considering their suitability as tracers for tire wear.

Table 2.5: Selected benzothiazoles and their characteristics in this study

	OHBT	**MBT**	**ABT**	**BT**	**MTBT**
CAS nr.	934-34-9	149-30-4	136-95-8	95-16-9	615-22-5
Traffic and road related sources	Rubber tire[1] Antifreeze[5]	Rubber tire[2] Antifreeze[6]	Rubber tire[3]	Rubber tire4 Antifreeze[2]	Rubber tire[1]
Aquatic toxicity	Cytotoxic[7]	Low toxicity[8]	Not toxic[3]	Acute Toxic[9]	Acute toxic[9]
Volatile in freshwater	Low[7]	Low[7]	Low[7]	Discrete[4]	Low[7]
Sediment adsorption coefficient ($\log Kow$)	2.35[10]	1.38[11]	2.00[10]	2.17[10]	3.22[10]
Water solubility (25°C)	2354 mg/L[12]	120 mg/L[12]	310 mg/L[12]	4300 mg/L[12]	125 mg/L[12]

[1]Kloepfer et al. 2005, [2]Wenderoth et al. 2004, [3]F. Zeng et al. 2016, [4]Reddy and Quinn 1997, [5]Asheim 2018, [6]Greaney 1995, [7]Liao et al. 2018, [8]Nielsen et al. 2014, [9]Reemtsma et al. 1995, [1]0Ye Yan and Weiwei et al. 2014,, [1]1Brownlee et al. 1992, [1]2Kirouani-Harani 2003

2.9.1 Soil/sediment absorption coefficient

LogKow can be an important factor for predicting distribution and the presence of a substance in the environment. Substances with high logKow values may tend to adsorb more readily to soil or sediments, because of their low affinity for water (Table 2.5). Chemicals with very high logKow values (>4.5) may be of great concern, thus they may potentially bioaccumulate in living organisms (Chemsafety pro 2016). MTBT has the highest logKow, making it the most hydrophobic of the selected benzothiazoles, while MBT is the least hydrophobic with its low logKow (Table 2.5). MBT is highly pH dependent, and according to Brownlee et al. 1992, its logKow value decreases with higher pH (Brownlee et al. 1992).

2.9.2 Degradation

Several studies investigate the microbial degradation of benzothiazoles in biological wastewater treatment plants (Reemtsma 2000, De Wever and Verachtert 1997, Gaja and Knapp 1997). The behaviour of benzothiazoles are often studied in pure- and mixed bacterial cultures, which are not expected to occur in such high concentrations in the environment, nor in tunnel wash water. In the environment, benzothiazoles are known to undergo biological, chemical and photolytic degradation, and these degradations are affected by pH, chloride and UV (Brownlee et al. 1992, (Kloepfer et al. 2005, Asheim 2018, Liao et al. 2018). The transformation cycle of BTs are comprehensive, thus they tend to transform several steps, and may even transform back to its origin compound (Brownlee et al. 1992) (Liao et al. 2018). Some studies show that BT (Reemtsma et al. 1995), MBT (De Wever and Verachtert 1997) and OHBT (Reddy and Quinn 1997) may mineralize and BT can possibly volatize in natural environment (Brownlee et al. 1992, Reddy and Quinn 1997). MBT is highly unstable and will decrease rapidly in aqueous solutions in the environment (Brownlee et al. 1992) (Kloepfer et al. 2005). MBT may undergo degradation through photolysis to OHBT (Felis et al. 2016) and BT (Brownlee et al. 1992), and may often biomethylate to the more stable MTBT, thus MTBT may not undergo further degradation in the environment (De Wever et al. 2001, Reemtsma et al. 1995). BT may degrade to OHBT through oxidation (Brownlee et al. 1992) and is also found to transform microbially to OHBT in settling ponds for road runoff (Reddy and Quinn 1997). Data on ABT degradation are scarce, but it is proposed that it will mineralize with the presence of chloride (Nika et al. 2017, De Wever et al. 2001) (e.g. road salt) and, to our far knowledge, ABT will not undergo further degradation to other thiazoles in natural environment.

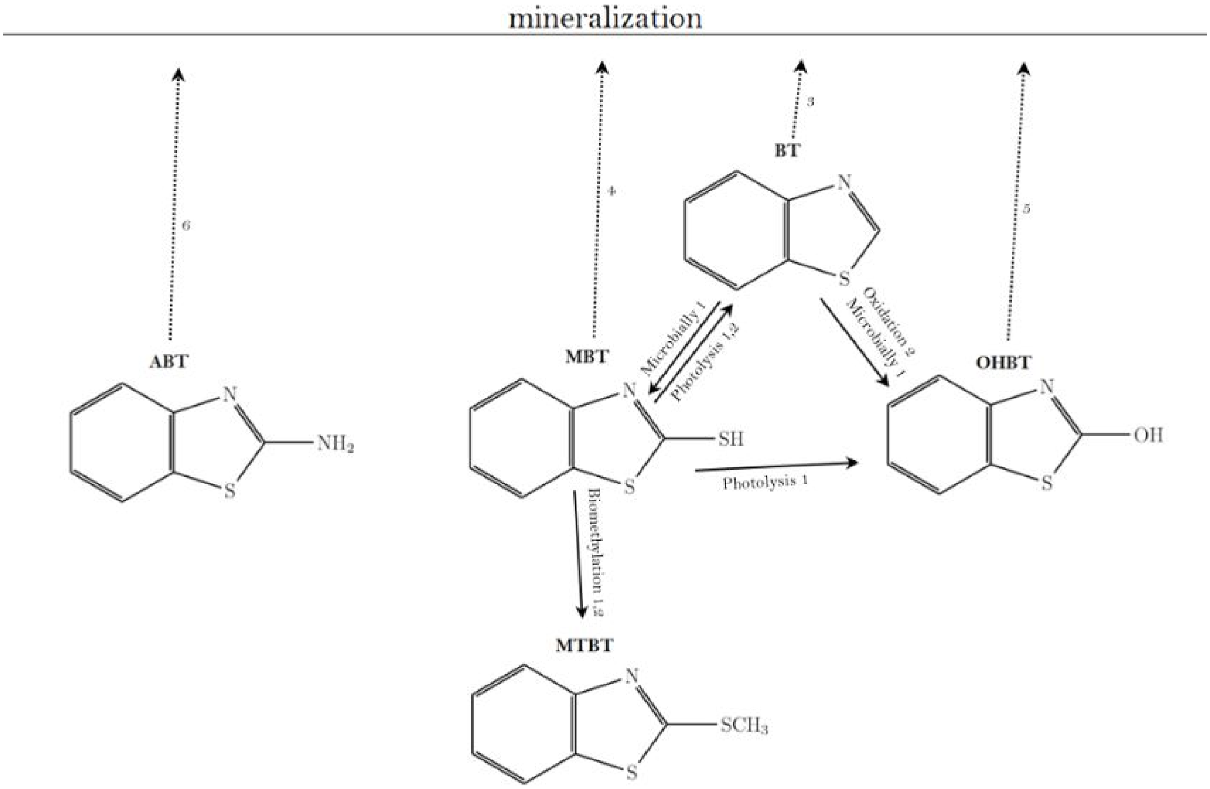

Figure 2.10: Degradation paths of the selected benzothiazoles according to [1]Felis et al. 2016, [2]Brownlee et al. 1992, [3]Reemtsma et al. 1995, [4]De Wever and Verachtert 1997, [5]Reddy and Quinn 1997, [6]Nika et al. 2017 and [7]De Wever et al. 2001

2.9.3 Solubility

MBT is more soluble in acid than in water (SCIENTIFIC COMMITTEE 2005). Due to their high water solubility, they are not believed to bioaccumulate (Liao et al. 2018). BT possesses a high solubility of 4300 mg/L (Kirouani-Harani 2003) and 3000 mg/L (Brownlee et al. 1992). This is probably due to the high polarity of BT and the fact that it is liquid at room temperature (20 °C) (Kirouani-Harani 2003). Temperatures can affect the solubility of the different BTs in various amounts, e.g. for MBT the solubility at 5 °C was about 40% lower than at 24 °C (Brownlee et al. 1992).

2.9.4 Leakage from tire particles

Leached benzothiazoles from tire debris, may be useful indicators for urban runoff and markers to quantify TWPs in the environment. It is estimated that benzothiazole (BT) and its derivatives (BTs) will leach from TPs in aquatic environments, and that the efficiency of leaching is dependent on particle size. Reddy and Quinn 1997 stated that ordinary TP with diameter <100 μm, can probably leach >50% of its BTs in water with pH of 5 (Reddy and Quinn 1997, Kumata et al. 2002). The leaching equilibrium of BTs from TPs will find place after 20 hours in water (Reddy and Quinn 1997), and is expected to occur in field (Hartwell et al. 2000). The size distribution of TPs used in the study by Reddy and Quinn 1997 is similar to the TWPs expected to be found in the environment(Hartwell et al. 2000), which indicates that the TP leaching process in field may be of similar rate, assuming the same composition of rubber as used in the study of Reddy and Quinn 1997.

2.9.5 Concentrations of Benzothiazoles in tire particles

In Table 2.6, the concentrations of BTs in TPs of passenger cars have been listed. The data show quite a large gap between the different authors, respectively from Norway and Sweden. This may be of geographical reasons or methods used to extract BTs from tires.

Table 2.6: Concentrations of benzothiazoles in different tires (ng/g)

Matrix	**OHBT**	**MBT**	**ABT**	**BT**	**MTBT**	**Ref.**
Summer tire		12 300		23 500	460	Avagyan et al. 2013
Summer tire		554 000-1 380 000**		39 300-155 000**		Avagyan et al. 2014
Summer tire	847	9 541	4.5	6 173	111	Asheim 2018
Winter tire		330 000		55 100		Avagyan et al. 2014
Stud free tire	2 229	21 407	3.9	13 807	197	Asheim 2018
Studded tire	1243	5 038	2.5	3 847	134	Asheim 2018
Studded tire		53 300-2 170 000**		60 300-78 300**		Avagyan et al. 2014
Rubber tire				6 160*	185 000*	Llompart et al. 2013

*Mean

**Low mean−high mean

3. Material and methods

3.1 Site description

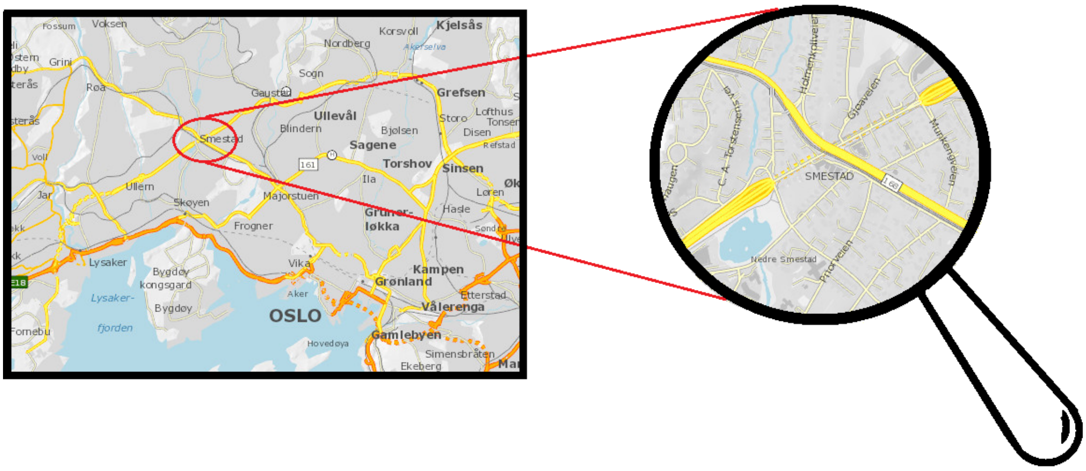

Figure 3.1: Site of study in Oslo, Norway. Zoomed in at sampling site Smestad tunnel. Statens Vegvesen 2019

The Smestad tunnel of 495 m is a located at the west side of Oslo (59°56’16” N, 10°41’2” Ø), on RV 150/Ring 3. It has two runs, two lane west going and two lane east- going. The building material is concrete elements on the wall and fire plates in the ceiling. The annual average daily traffic (AADT) was measured to 57 888 vehicles per day in 2018 (Statens Vegvesen 2019). The tunnel is washed as scheduled in Table 2.4 and has a treatment system for tunnel wash water containing a drainage system, pumping station, sedimentation basin and a rain garden, as shown in Figure 3.2. Under a "normal situation", the sedimentation basins are closed because they are constructed for tunnel wash water only. This means, when stormwater is entering the treatment system, it is pumped directly to the rain garden for infiltration as the only treatment.

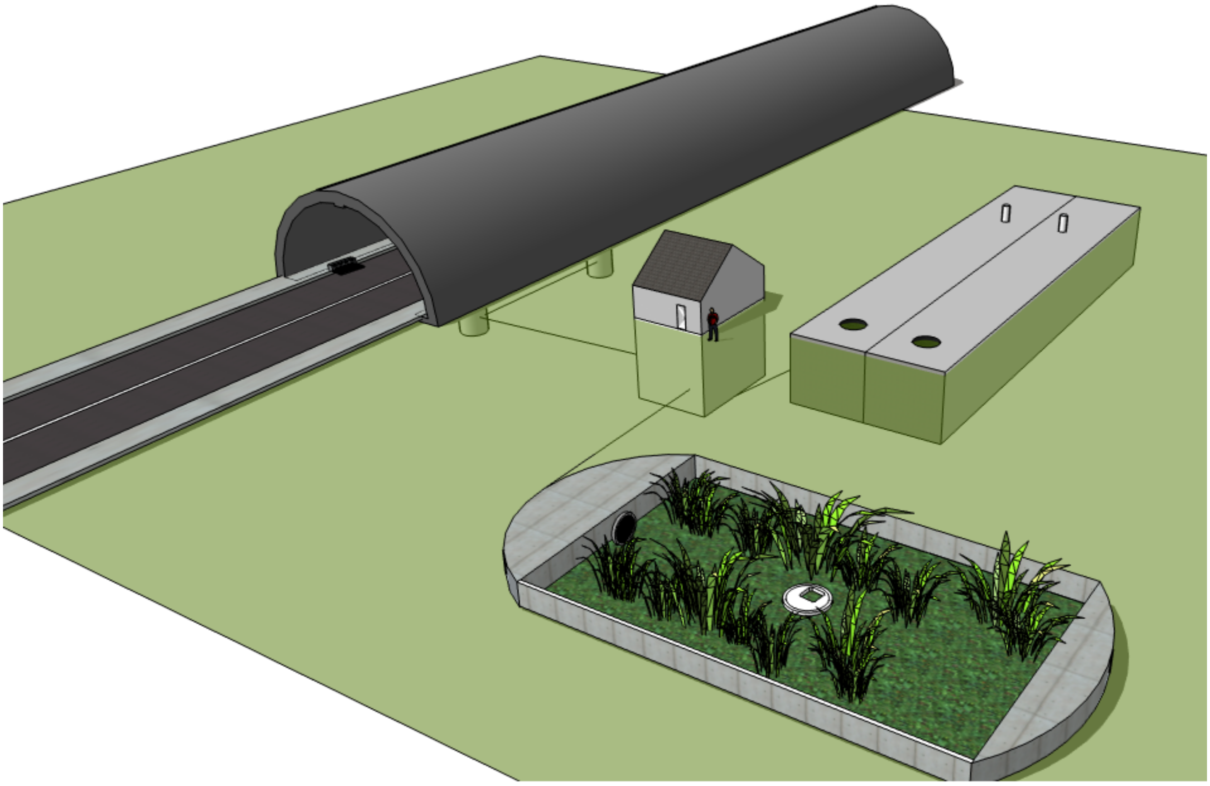

Figure 3.2: Conceptual drawing of Smestad tunnel with corresponding treatment process.

Drainage system

The westbound tunnel has 18 gully pots with submerged outlet for restraining floatable contaminants. (Appendix C.1 and C.2).

Pumping station

The pumping station consist of a pump sump, two level controlled pumps, valves and a overflow to the Smestad pond. The pump stations function is to pump the tunnel wash water or the stormwater runoff to the associated sedimentation chamber and/or the rain garden. The pump sump is emptied for sludge and cleansed twice a year.

Sedimentation basin

The sedimentation basin consists of two large separate chambers underground which are connected to the westbound and the eastbound tunnel respectively. Each chamber has a pipe leading water from the pump station and a pipe leading the water to the pump station by gravity, after the sedimentation. The gravitational pipe flow are controlled by automatic valves which reopens three weeks after the tunnel wash. The basin also has a overflow to the Smestad pond. Sludge removal and cleansing of the sedimentation basin are performed once a year. (Appendix E).

Rain garden

The rain garden receives stormwater or sedimentation water from the pump station via an inlet grate. The rain garden are constructed as a flower bed with a granite frame and a top layer of organic material where accumulation of pollutants occur. The water infiltrates through layers of various filter media and is collected on a geotextile covering half of the construction, making the water available for sampling in the manhole placed in the center of the rain garden. In the other half the water can infiltrate to the ground. Excessive water is led through a drainage pipe to the Smestad pond. Maintenance is performed once a month from May to October, and the procedure includes weeding, reestablishment of the organic top layer and plants and inspection of the outflow to the recipient. (Appendix E).

3.2 Technical wash sequence

Preparing the tunnel wash

The day before the washing event the entrepreneur controlled that automatically emptying of the sedimentation basin was started. According to the wash plan of Norwegian Public Roads Administration the washing entrepreneur were supposed to start the program of forced emptying the sump, which was not done November 5^{th} , and the sump contained 30 m^3 of stormwater runoff before the wash.

Before the westbound tunnel with associated lanes were washed, the road and pavement were swiped for dust and grit. There was no rain event one week before the tunnel wash.

During the tunnel wash

The entrepreneur switched the PLC (Programmable logic controller) from normal situation to washing situation for the west bound tunnel. The tunnel wash initiated with soap-containing high pressure wash of ceiling and technical gear, followed by walls and road signs, and finished off with the road surface and pavement. Later, in the same order, the tunnel was pressure washed with clean water. In total, 25 m^3 water and 30 liter of soap were used during the wash. This gives a consumption of approximately 50 L/m of tunnel. The wash water was pumped to the west bound associated sedimentation chamber at 7 L/s.

After the tunnel wash

The entrepreneur switched the PLC back to normal situation, and the valves controlling the flow from the sedimentation basin were closed, and a countdown for reopening was automatically set to 2 weeks and 6 days. After sedimentation the wash water was led to the pump sump. In this case, as well as when the tunnel wash initiated, the sump was not emptied for stormwater water before receiving tunnel wash water. Meanwhile the sump received water from the west bound associated sedimentation chamber, a pump started pumping water at a flow rate of 1.5 L/s to the rain garden for infiltration.

3.3 Sampling of environmental samples

To assess the amount of TWPs entering the treatment processes, samples of the wash water runoff were taken and sent for detection of BTs concentration. The concentrations were then compared with concentrations in TPs. The concentration of BTs throughout the whole treatment system was investigated in order to assess the treatment efficiency of TPs. A total of 22 samples were collected in Smestad tunnel and its corresponding treatment facilities for tunnel wash water. To study degradation and transformations of the tracer compounds (BTs) over time, the concentration of BTs in sedimentation basin was sampled over three weeks, samples of sediments were taken and behaviour during storage at laboratory was assessed.

Plastic bottles for sediment samples were filled 1/2 and liquid samples were filled 2/3. The sediment samples were collected using a Van Veen Grab, except for the top soil samples which were collected using a plastic spoon.

Sampling sites before wash

Sediment samples were collected from three gully pots (Figure 3.9, point S1, and Appendix C) before wash. The three selected gully pots were those expected to receive the greatest amount of wash water based on their low elevation. Mixed samples (three samples) were taken in each gully pot, then placed on a metal plate and stirred with a plastic spoon (Polystyrene) before it was collected in sampling bottles.

The topsoil in the rain garden (Figure 3.9, point S3) was also sampled before the wash water entered the system to study the presence and distribution of the BTs in the top layer. Topsoil was collected at three different sites near the inlet and as for the gully pot sediment samples, the sediment collection was stirred to give a well mixed sample.

Sampling sites during wash

The first samples collected during the tunnel wash were the runoff samples, collected directly from the carriageway (Figure 3.9, point W1). To make the sampling more efficient, a bucket was lowered down inside the gully pot to catch the runoff water before it entered the drainage system. To give representative and mixed samples, the bucket was "rinsed" in the runoff water once, then filled with small amounts over time. The sampling bottle were “rinsed” in the bucket water three times before the final sample was collected. This procedure was performed at all of the three selected runoff locations, which were the same sites as for the gully pot sediment samples before the wash (Appendix C).

The pump started automatically when the level in the sump reached 2.37 m and it started to fill the sedimentation chamber of the westbound tunnel at a flow rate of 1.7 L/s. Samples were taken at three different times during the wash; at the beginning, in the middle and in the end of the washing process. Each sample was taken in a sunken bucket of 10 liters, witch was “rinsed” in the wash water once, then filled. The sampling bottles were “rinsed” in the bucket water three times before the final sample was collected. This were done for all three samples (Figure 3.9, point W2).

Figure 3.3: Sampling of wash water runoff. Photo: Sondre Meland, NIVA.

Figure 3.4: pumping chamber with foaming soap during tunnel wash. Photo: Ninni Bye.

Sampling sites after wash

In total, three mixed samples were taken from the sedimentation chamber (Figure 3.9, point W3) of the west bound tunnel after one, two and three weeks respectively. The sampling procedure was equal the previous liquid sampling procedures; a bucked was “rinsed” once in the stored wash water, then filled. The sample bottles was “rinsed” in the bucket water three times before the final sample was collected. The same procedure were repeated each week, for three weeks.

When the sedimentation chamber was emptied there were taken two samples of the sediments in the chamber (Figure 3.9, point S2), one as a liquid sample of the "sludge", and one coarse sample as sediment for liquid extraction in laboratory. The day after sampling in the sedimentation basin, the "clean" water was let by gravity through an open valve into the pump sump.

Figure 3.5: Collection of mixed sample from sedimentation basin. Photo: Jon Petter Johnsen.

Figure 3.6: The rain garden. Photo: Ninni Bye.

The sedimented wash water were pumped to the rain garden inlet (Figure 3.9, point W4) at 1.5 L/s in batches run by the level in the pump sump. In total three mixed samples were collected at the start, the middle and in the end of the discharge, according to the PLC screen showing the water level in the sedimentation chambers. The sampling bottle was “rinsed” three times with inlet wash water, then filled. This was done for all three samples. The sedimentation chamber was emptied over a total of 8.5 hours.

Figure 3.7: Treated wash water discharged from the sedimentation basin to the rain garden. Photo: Jon Petter Johnsen.

In total three mixed samples were collected using a sampling device to scoop up infiltrated water form the manhole (Figure 3.9, point W5). As the water had infiltrated through the rain garden, and the manhole was filled, the first sample was collected. The two remaining samples were collected when the second and third round of inlet water was expected to have filled the manhole, a couple of hours after the respective inlet. The estimated infiltration time were very speculative and were assumed to give an approximate representative sample. The sampling bottles were “rinsed” with the infiltrated water three times before the final sample was collected. This were done for all three samples.

Figure 3.8: Sampling in the rain garden man hole. Photo: Ninni Bye.

The topsoil in the rain garden (Figure 3.9, point S3) was sampled with the same procedure as before the wash. Topsoil was collected on a metal plate and taken from various location sites around the inlet. The soil was mixed well before a final sample was taken.

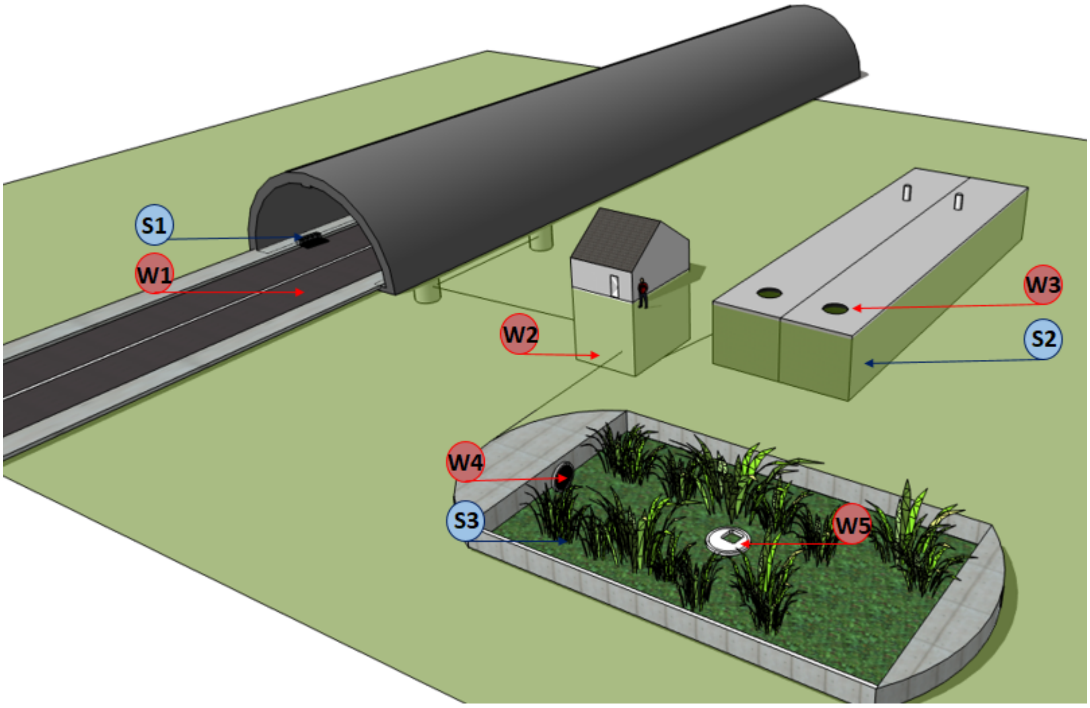

Figure 3.9: Sampling sites for sediment (blue circle) and water (red circle).
S1=gully pot, S2=sedimentation basin, S3=top soil. W1=runoff, W2=pump sump, W3=Sedimentation basin, W4=rain garden inlet, W5=rain garden manhole.

Sample storage

Immediately after the samples were taken they were delivered at the laboratory at NIVA for cryopreservation in −20 °C. They were stored for a month before the first selection of samples were analyzed January 31^{st}. The rest of the samples were analyzed after one month of storage in a fridge (4.5 °C). The sediment samples were stored 12 days in the fridge before analysis. All the analysis were performed by Jan Thomas at NIVA and is further described in Appendix D.

3.4 Calculating TP emission

The methodologies for estimating the TP emission in the Smestad tunnel are presented in Figure 3.10. The use of concentrations of BTs in tunnel wash water runoff are dependent on the percentage leakage of BTs from TPs in addition to the concentration of BTs in TPs, which varies in different studies. The estimates of daily TP emission will vary accordingly. Equation 3.1 is dependent on choice of emission factor which varies in different studies, leading to variations in estimated TP emission by this method as well. The two methods of estimation will be compared to each other, in addition to other studies.

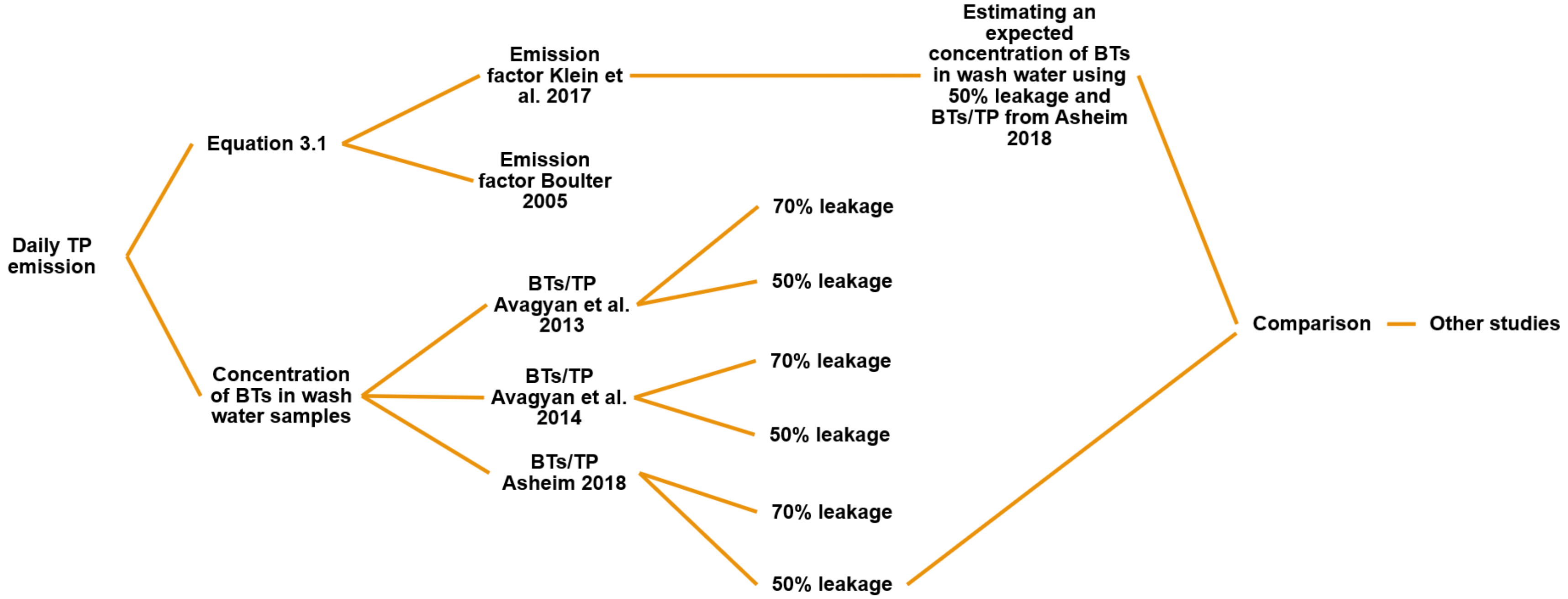

Figure 3.10: Methods for estimation of daily TP emission in the Smestad tunnel.

3.4.1 TP emission based on concentrations of BTs

To be able to create a methodology for calculating the TP emission present in tunnels and tunnel wash water, there are numerous important factors to consider when using the concentrations of benzothiazoles, illustrated in Figure 3.11. Understanding the behaviour of BTs in water, transportation paths of TWPs and performance of tunnel wash is crucial in order of determine the validity of the calculations and suitability of BTs as tire wear tracers.

The calculation method initiates with the detected concentrations for each of the BTs, in the wash water runoff samples. Furthermore, the concentrations are multiplied with the amount of wash water used, in order of detecting the mass of the BTs instead of concentrations. The removal of particles needs to be considered at this step, since main part of the leached BTs happens when the TPs are exposed to water. Therefore an addition of the particles assumed removed due to sweeping before wash needs to happen. Furthermore the particulate bound BTs, which are not leached from particle, meaning not detected in samples, needs to be added. At this step the initial mass of BTs before wash and sweeping are calculated. The next step is to divide the initial mass on the concentration of BTs in TPs, using concentrations detected from suitable studies depending on e.g. winter/summer tires, selection of studied BTs etc. The calculations are resulting in the mass of TPs present, according to the concentration of BTs. If accumulation time is known, divided with number of days will result in daily TP emission.

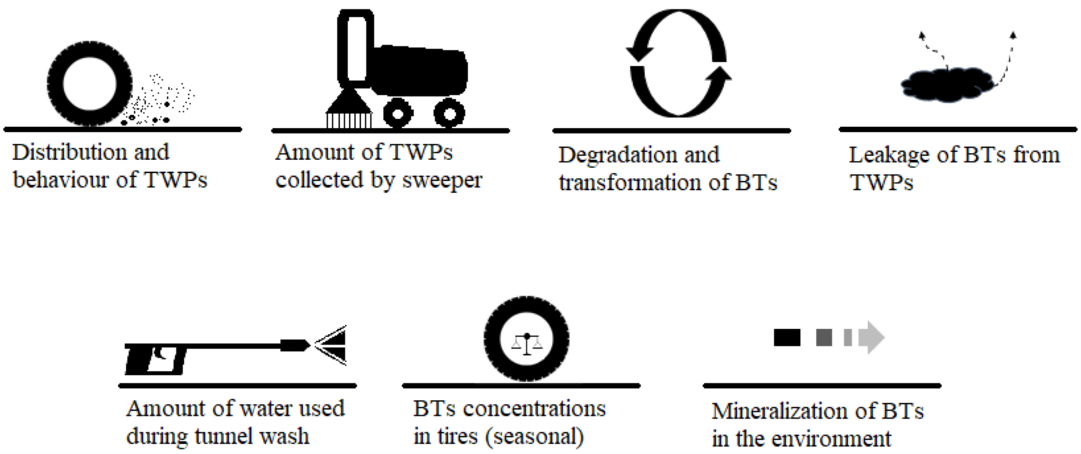

Figure 3.11: Important factors to consider when calculating TP emission in road tunnels based on BTs concentrations in tunnel wash water.

Meland and Roseth 2006 found that 20−80% of tunnel contaminants were removed by suction/sweeping. Based on this study, an assumed 50% removal due to suction and sweeping were used in the calculation of estimated concentrations in Table 4.1. Secondly, the amount of particulate bound BTs had to be subtracted in the calculations because the restrained BTs could not be detected in the laboratory. As stated by Reddy and Quinn 1997, the small TPs could probably leach >50% BTs in water with pH of 5. For the BTs, a leakage of 50% and 70% respectively, has therefore been used to estimate the results. Tunnel wash water is expected to have a pH of minimum 7.4, dependent on soap concentrations (Aasum 2013), and the percentile leakage of BTs at these pH values are unknown.

To estimate the amount of BTs emitted in the Smestad tunnel, concentrations of BTs in tread of summer tires from the previous studies of Asheim 2018 and Avagyan et al. 2013 and Avagyan et al. 2014, were chosen from Table 2.6. and presented in Table 3.1. It was assumed that the actual TP emission originated from summer tires, giving the time period of sampling (studded tires are permitted in Norway from November 1^{st}). The TP emission has been accumulating in the tunnel since the last tunnel wash (60 days).

Table 3.1: Concentration of BTs in summer tire particles detected by Asheim 2018 and Avagyan et al. 2013 and Avagyan et al. 2014. Concentrations in ng/g.

	OHBT	**MBT**	**ABT**	**BT**	**MTBT**	**ΣBTs**
Asheim 2018	847	9 541	4.5	6 173	111	16 675
Avagyan et al. 2013	n/a	12 300	n/a	23 500	460	n/a
Avagyan et al. 2014	n/a	554 000-1 380 000*	n/a	39 300-155 000*	n/a	n/a

*Low mean−high mean

3.4.2 TP emission based on equation

To compare the calculated daily TP emission based on concentration of BTs, the Equation 3.1, by Vogelsang et al. 2019, are suggested as a comparable reference.

$$E_{T,r,t} = \sum\ _{r,i}(L_r \cdot N_{r,i,t} \cdot EF_{i,j}) \tag{3.1}$$

- $E_{T,r,t}$ is the total tread emission along the road stretch *r* over a given time period *t* (mg).
- L_r is the length of the particular road stretch *r* (km).
- $N_{r,i,t}$ is the number of vehicles in category *i* that have travelled the particular road stretch r during the given time period *t* (AADT).
- $EF_{i,j}$ is the specific tread emission factor for vehicles in category *i* for the relevant type of driving (mg/vkm).

4. Results and discussion

4.1 Calculation of TP emission

Estimated daily TP emission in the Smestad tunnel based on detected concentrations of BTs in wash water runoff

Table 4.1 shows a calculation examples of daily TP emission in the Smestad tunnel, when considering 50% removal by sweeper and 50% leakage of BTs from the TPs. Furthermore, the calculation example are using the concentration of BTs in TPs from Asheim 2018. The Table does not take aquatic behavior of BTs into consideration (e.g. degradation and possible variations in leachability from particles).

Table 4.1: Calculation example of daily TP emission in the Smestad tunnel.

	OHBT	**MBT**	**ABT**	**BT**	**MTBT**	**ΣBTs**	Unit
Concentration in wash water	1427	<1	28	15343	224	17021	ng/L
Resulting mass [1]	35.675		0.7	383.575	5.6	425.525	mg
Initial mass [2]	0.143		0.003	1.534	0.022	1.702	g
TP emission [3]	168.477		623.608	248.550	201.802	102.075	kg TPs
Daily TP emission [4]	2.8		10.4	4.2	3.4	1.7	kg TPs /day

1. Dry Weight of leaked BTs present in wash water after sweeper.
2. Expected dry weight of BTs present in the tunnel before the washing event.
3. Total TP emission accumulated after previous tunnel wash.
4. Total daily TP emissions.

To compare the calculated daily TP emissions when considering 50% leakage, a chosen value of 70% leakage from particles are used in order to clarify the uncertainties concerning leakage of BTs from tires in Table 4.2. Reddy and Quinn 1997 stating that small TPs probably leach >50% BTs in water with pH 5, and to our far knowledge there are no literature considering leakage of BTs in tunnel wash waters affected by soap, oil or high pH.

Table 4.2: Calculated emissions in kg TPs/day when using concentrations of BTs in tires from three studies of summer tires (from Table 3.1), and assumed 50% leaching of BTs from TWPs. compared to 70% leaching

		OHBT	**MBT**	**ABT**	**BT**	**MTBT**	**ΣBTs**
50% leakage	Asheim, 2018	2.8	–	10.4	4.2	3.4	1.7
	Avagyan et al. 2013	n/a	–	n/a	1.1	0.8	n/a
	Avagyan et al. 2014	n/a	–	n/a	0.2–0.7*	n/a	n/a
70% leakage	Asheim 2018	2	–	7.4	3	2.4	1.2
	Avagyan et al. 2013	n/a	–	n/a	0.8	0.6	n/a
	Avagyan et al. 2014	n/a	–	n/a	0.1–0.5*	n/a	n/a

*Low mean–high mean

When comparing the calculated daily TP emission using the concentrations of BTs in TP for different literature (Asheim 2018, Avagyan et al. 2013, Avagyan et al. 2014), it shows that variations in concentrations of BTs in tires may have large impact on the results. This gives great uncertainties when estimating the TP emission by the concentration of BTs. The large differences in content of BTs in tires may be due to differences in composition when manufacturing, or the method of analysis in laboratory. These results indicate that leakage of BTs from TPs will generate large uncertainties in the calculations of TP emission. By adding 20% more leakage, the estimated TP emission decreased by almost 1 kg TPs/day inside the westbound tunnel. In order to reduce the uncertainties due to leakage, more research on BTs concentration in tires is advised.

Estimated TP emission based on Equation 3.1

Table 4.3 contains values for length of the tunnel, the AADT for passenger cars and long vehicles published by Norwegian Public Roads Administration at Statens Vegvesen 2019 for the year 2018. The emission factor for each of the vehicle categories was chosen from Table 2.3. The traffic in the Smestad tunnel categorized as highway driving style, due to relatively smooth traffic flow and high speed. In the rush hours it would occasionally categorize as urban driving style. The highway driving style was used for parameter $\mathrm{EF}_{i,j}$. To calculate the estimated amount of TP emission accumulated on the road each day in the time period since the previous tunnel wash, the Equation 3.1 was utilized.

$$E_{T,r,t} = \sum\ _{r,i}(L_r \cdot N_{r,i,t} \cdot EF_{i,j}) \tag{3.1}$$

This resulted in an estimated emission of 3.3 kg each day during the two month period.

Table 4.3: Parameters in TP emission Equation 3.1, with corresponding values for the Smestad tunnel.

Vehicle category	**L_r (km)**	**$\mathrm{N}_{r,i,t}$ (AADT)**	**$\mathrm{EF}_{i,j}$ (mg/vkm)**	**E_T (kg/day)**
Passenger car	0.5	46 500	104	2.42
Heavy duty vehicle	0.5	3 500	504	0.88
Total TP emission				3.30

$\mathrm{EF}_{i,j}$ from Table 2.3, Klein et al. 2017

AADT from Statens Vegvesen 2019

To show how the choice of emission factor $\mathrm{EF}_{i,j}$ affects the resulting daily TP emission when using Equation 3.1, the $\mathrm{EF}_{i,j}$ values of Boulter 2005 are replaced with the $\mathrm{EF}_{i,j}$ used in Table 4.3. For passenger cars, Boulter 2005 suggested an TP emission of 100 mg/vkm for passenger cars, and 1000 mg/vkm for heavy- duty vehicles, for "normal" driving conditions. Using the same calculation method as for Table 4.3, the estimated daily TP emissions are 4.1 kg/day.

Equation 3.1 is expected to give a good estimate for daily TP emission, but there are numerous parameters required to perform such a calculation. There are not always easy to obtain information like AADT and emission factors out in the field. This is why it is of great interest to find other tracers for detecting the presence of TP in the environment.

Estimated concentrations of BTs based on Equation 3.1

Now it is possible to estimate the daily emission for each of the BTs. Total daily TP emission is calculated in Table 4.3, using Equation 3.1, giving a resulting daily TP emission the range of 3.3 – 4.1 kg TPs/day. When using the mass emission in further calculations in Table 4.4, 3.3 kg TPs/day is assumed to give the best estimate, since the emission factors from Klein et al. 2017 are considered to adjust to the driving conditions in the Smestad tunnel better than the emission factors of Boulter 2005. However, the TP emission given by the latter are represented in Figure 4.1 to illustrate uncertainties. The BTs /TP concentration of Asheim 2018 (Table 3.1), are the only studies investigating all of the BTs relevant in this thesis, and his study are the one used in the estimations shown in the tables below. Emission of BTs and the accumulated BTs over a 60 day period are calculated in Table 4.4.

Table 4.4: Emission of BTs per day in the Smestad tunnel and the resulting mass of BTs accumulated during the 60 day period after previous tunnel wash.

	OHBT	**MBT**	**ABT**	**BT**	**MTBT**	**ΣBTs**
Daily emission[1] (g/day)	0.0028	0.032	0.000015	0.021	0.00037	0.055
Accumulated in 60 days (g)	0.17	1.89	0.00089	1.22	0.022	3.302

[1] Concentration of BTs from first row of Table 3.1 multiplied by the total TP emission from Table 4.3.

The mass of BTs emitted in the tunnel were exposed to 25 000 liters of wash water. The estimated concentrations of BTs per liter wash water runoff, based on Equation3.1, are presented in the first row in Table 4.5.

To compare the estimated concentrations of the BTs based on Equation 3.1 to the actual concentration in the field samples, adjustments due to sweeping and leakage had to be done as shown in Table 4.5. The lower row in Table 4.5 presents the mean concentrations of the three wash water runoff samples. The concentrations in bold are comparable and useful when investigating the various BTs' suitability as markers for TWPs in the tunnel, and for discussion of transformation/degradation. In Table 4.5, as for Table 4.1, the estimated concentrations based on Equation 3.1 does not take the aquatic behavior of the BTs into consideration (e.g. degradation and variations in leachability from particles).

Table 4.5: The concentration of BTs in wash water runoff estimated from Equation 3.1, subtracted by 50% when considering particle removal by sweeper and another 50% subtracted when considering 50% leakage of BTs from TPs. Concentrations in ng/L.

Concentrations	**OHBT**	**MBT**	**ABT**	**BT**	**MTBT**	**ΣBTs**
Based on Equation 3.1	6 708	75 564	35	48 890	880	132 077
Considering 50% removal by sweeper	3 554	37 782	18	24 445	440	66 039
Also considering 50% leakage from TP	**1 777**	**18 891**	**9**	**12 223**	**220**	**33 020**
Measured in wash water runoff	**1 427**	**<1**	**28**	**15 343**	**224**	**17 021**

A benzothiazole that stands out in the table is MBT, which is estimated in Table 4.5 to have a concentration of 18 891 ng/L, while the detected concentration is <1 ng/L. MBT is highly unstable and will decrease rapidly in aqueous solutions in the environment (Brownlee et al. 1992, Kloepfer et al. 2005, E. Zeng et al. 2004). The low detection of MBT in the wash water runoff samples can be caused by mineralization (Figure 2.10) (De Wever and Verachtert 1997). Other transformation routes of MBT are by photolysis to OHBT (Felis et al. 2016) and BT (Brownlee et al. 1992), but occurrence of photodegradation is unlikely, since the tunnel wash water is not exposed to direct sunlight. Another possible transformation process is biomethylation by microorganisms to MTBT (De Wever et al. 2001, Reemtsma et al. 1995), but since the MTBT concentration in the field sample correspond to the estimated concentration in Table 4.5, may be explained by biomethylation from MBT to MTBT have occurred, but the concentrations have decreased during time in fridge (further explained in Chapter 4.2). Another explanation may be that biomethylation may not be of significant impact. The latter explanation are supported by the study of De Wever et al. 2001, showing that biomethylation of MBT to MTBT occurred in low yield. Another possible cause may be Transformation from MBT to other BTs which are not investigated in this study can also be the cause, or challenges with detection at laboratory. OHBT, ABT, BT and especially MTBT shows concentrations as expected when compared to estimated calculations, considered a large amount of uncertainties in many steps of the calculations e.g. fitness of Equation 3.1, mass of TP emission per vehicle km (only based on driving location and vehicle type), and major uncertainties related to the removal of BTs due to sweeping, and varieties in leakage of BTs from particulate phase.

Uncertainties linked to the wash water runoff samples of this study may be due to other possible contributing sources of BTs, like spillage of antifreeze, larger fractions of tire debris or garbage. The runoff concentrations of the BTs are mean concentrations detected from runoff on three different locations in the tunnel, assumed to only give an approximate representation of the concentrations entering the treatment system.

The detected concentrations of BTs are compared to earlier studies of BTs in storm water runoff, in lack of studies of tunnel wash water. The studies available can still give some comparable information. The concentrations of most of the BTs detected in this study of tunnel wash water runoff are higher than in stormwater runoff (Ni et al. 2008, Reddy and Quinn 1997, E. Zeng et al. 2004). E. Zeng et al. 2004 detected in 1997 – 1998 the following concentrations for BT and MTBT in stormwater runoff; 86 – 555 ng/L and 0 – 160 ng/L respectively. Reddy and Quinn 1997 detected stormwater runoff concentrations in the range of 378 – 1210 ng/L of BT, and 5000 – 7000 ng/L of OHBT in Rhode Island, USA 1995. It should be taken into consideration that the AADT in these literatures are unknown, and that AADT are an important factor affecting the emission of BTs (e.g. TPs).

The measured BT concentration in this study shows significantly higher concentrations of BT, 15 times higher than in regular stormwater runoff. This was expected when tunnel wash water are expected to be more concentrated by pollutions than stormwater runoff. The wash water concentration for MTBT of 224 ng/L were quite similar to those of stormwater runoff of 160 ng/L. The measured 1427 ng/L of OHBT deviates from the expectations, with a concentration 4 – 5 times lower than in stormwater runoff (5000 – 7000 ng/L), in addition to an expected contribution of OHBT due to degradation from BT. The differences in stormwater pH and tunnel wash water pH, may have influence on leachability and/or degradation of BTs, and can be a possible explanation of the differences in detected OHBT concentrations.

Comparison of the estimated TP emissions based on concentrations of BTs measured in wash water runoff samples, and estimations based on Equation 3.1.

The results of the two methods of estimating the daily TP emission in the Smestad tunnel are illustrated in Figure 4.1. Estimated daily TP emission for each of the BTs, based on concentration of BTs in the tunnel wash water runoff, can be compared to the the estimated daily TP emission from Equation 3.1. The estimated TP emission of 3.3−4.1 kg TPs/day, from the Equation, are values that represents the daily average TP emission over a two month period (approximately 60 days between previous tunnel wash and the wash November 5^{th}). The variations in the emission factor $\mathrm{EF}_{i,j}$ affecting Equation 3.1, are illustrated as a line between the resulting 3.3 kg TPs/day and 4.1 kg TPs/day. Regarding variations in the concentrations of BTs in TPs, in the studies of Asheim 2018, Avagyan et al. 2013 and Avagyan et al. 2013 are illustrated by different colors. The circle and star symbols, represents the estimations when considering 50% and 70% leakage of BTs from TPs respectively. The line connecting the symbols represents the range of expected TP emission in the actual range. This illustrates that a 20% change in leakage can affect the emission estimations, meaning relatively large uncertainties in these calculations.

When reading the graph in Figure 4.1, there is important to keep in mind that aquatic behavior of the BTs e.g. mineralization and degradation between the BTs, is not accounted for. To discuss the graph, knowledge regarding the behaviour of BTs (Chapter 2.9) is important.

Using the total concentration of BTs to estimate daily TP emission gave a very low estimate (1.2 – 1.7 kg TPs/day), compared to the calculated emission by Equation 3.1.This is probably due to the low detection of MBT (<1 ng/L) in the wash water samples (Table 4.5) and that its concentrations in tires are included in the calculation. MBT are therefor excluded in further investigations, and a new TP emission based on the sum of BTs is presented in 4.1. Using the sum of concentrations of OHBT, ABT, BT and MTBT and the same calculation method as explained i Table 4.1, and BTs/TP relations from Asheim 2018, the resulting daily TP emission are 2.8−4.0 kgTPs/day, estimating 70% and 50% respectively.

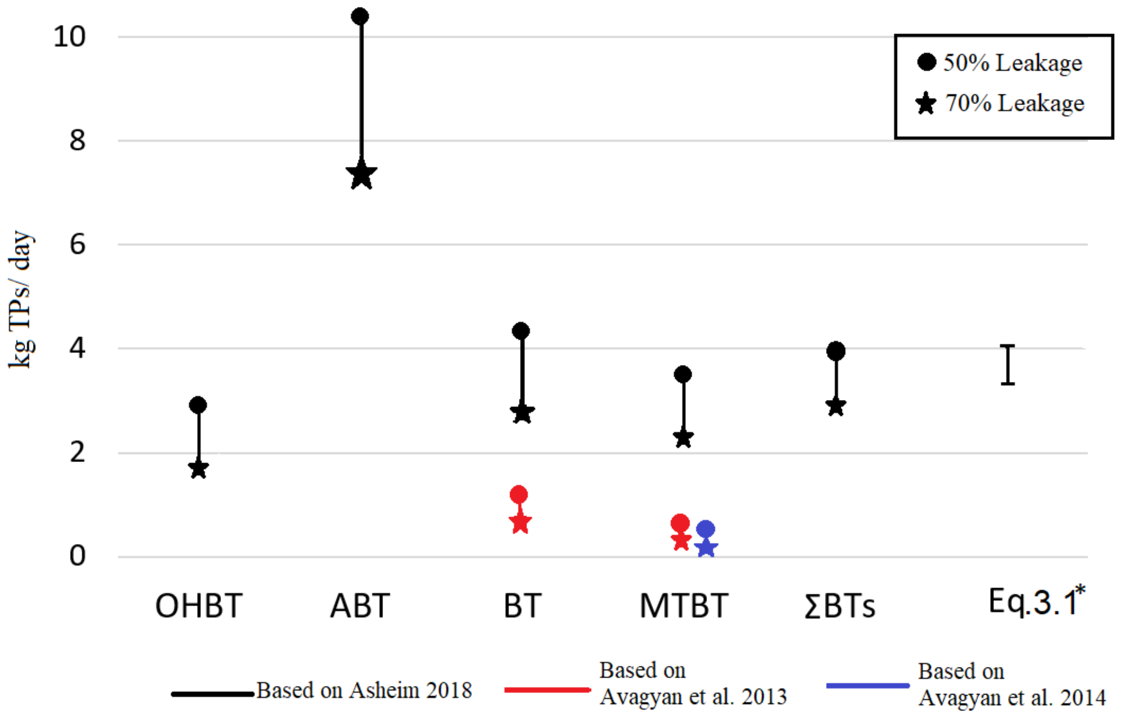

Figure 4.1: Daily TP emission calculated from concentrations of the selected BTs in the wash water runoff samples, with comparable values from Equation 3.1. Dots and stars represents the TP emission calculations with 50% and 70% leakage from particles respectively. The colors illustrates how choice of study regarding concentrations of BTs in summer tires effects the results.

* 3.3 – 4.1 kg using emission factors from Klein et al. 2017 and Boulter 2005 respectively.

4.2 Concentration of benzothiazoles in the treatment system

Figures 4.2, 4.3, 4.4 and 4.5, show each of the analyzed benzothiazoles throughout the whole treatment system and they were analyzed at laboratory in two rounds. The red column were analyzed first (January 31th), and the blue columns were analyzed one month later (February 25th), meaning the blue columns were analyzed after one month of storage at 4°C. For MBT, all of the analyzed liquid samples had concentrations <1 ng MBT/L and are therefore not illustrated in the graphs. The following illustrations will focus mainly on the retention and behaviour of the other BTs during the treatment process in the Smestad tunnel. To be able to discuss the restrain and distribution of TWPs, it is crucial to understand the behaviour of the BTs. The retention and distribution of TWP will be discussed in section 4.3.

OHBT

The reduction of OHBT from initial inlet to final outlet of the treatment system is around 85% (Figure 4.2). OHBT concentrations in the wash water increases after entering the pump sump, which may be explained by the degradation of BT and MBT to OHBT. Even with a high solubility of 2354 mg/L, the great reduction after one week of sedimentation is expected, thus OHBT has a high logKow value (Table 2.5). As the sedimentation process was ongoing, there was a rain event that filled the pump sump with stormwater runoff from the tunnel, this was later mixed with the sedimented/treated water before it was pumped to the rain garden. The dilution with untreated water may be the reason for the drastic increase of OHBT in the rain garden inlet. The first of the samples taken from the rain garden inlet was expected to be the most diluted by rain water, this could explain the high concentration compared to the second and third sample. Because the sedimentation samples were analyzed one month earlier than the rain garden inlet samples, the possible degradation from BT and MBT to OHBT may not have time to occur in the same degree as

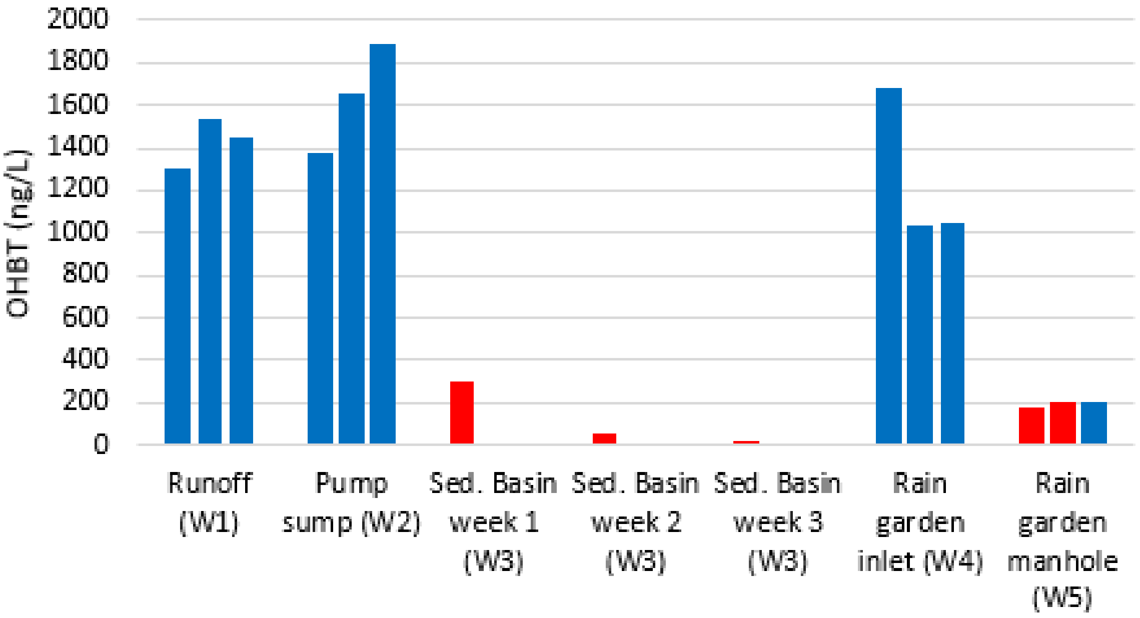

Figure 4.2: Concentration of OHBT throughout the treatment system. Red colored bars have been stored one month at 4°C.

if they were analyzed in February. If the sedimentation samples were analyzed in February, the increase between sedimentation basin week 3 and rain garden inlet would probably not be as drastic as shown. The three samples that were collected in the rain garden manhole, appears to be comparable. This may indicate that OHBT in the manhole samples does not undergo any remarkable degradation in the fridge. The diagram illustrates how OHBT settles in the sedimentation basins as well in Figure 4.6

MTBT

The data on MTBT throughout the treatment system is not as expected (Figure 4.3). As mentioned, the MTBT was expected to be stable and not to undergo any remarkable degradation. It is believed that the concentrations of MTBT were higher when the samples were collected, but that MTBT degraded and/or mineralized before the samples were analysed after one month in the fridge. This could also mean that time related degradation has occurred in the sedimentation basin. The degradation of MTBT is substantiated in Figure 4.7. The three samples from the sedimentation basin could also illustrate that MTBT is settling well, as expected due to the high logKow value of 3.22.

BT

The total treatment efficiency of BT, from inlet to outlet, is approximately 90% (Figure 4.4). This reduction may be reached by the treatment facility and by degradation of BT to OHBT. BT has the highest solubility of 4300 mg/L, which indicates that it is highly mobile in the water phase and may not settle remarkably. The small reduction in the sedimentation basin may be due to degradation to other thiazoles, like OHBT (Figure 2.10).

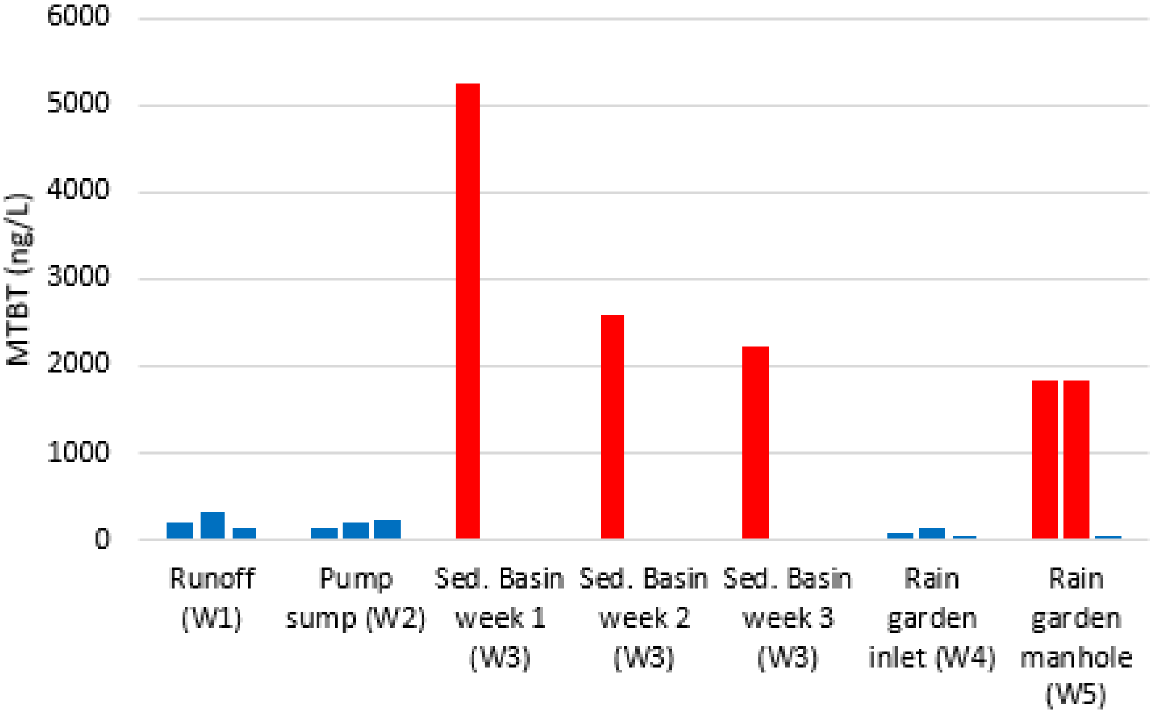

Figure 4.3: Concentration of MTBT throughout the treatment system. Red colored bars have been stored one month at 4°C

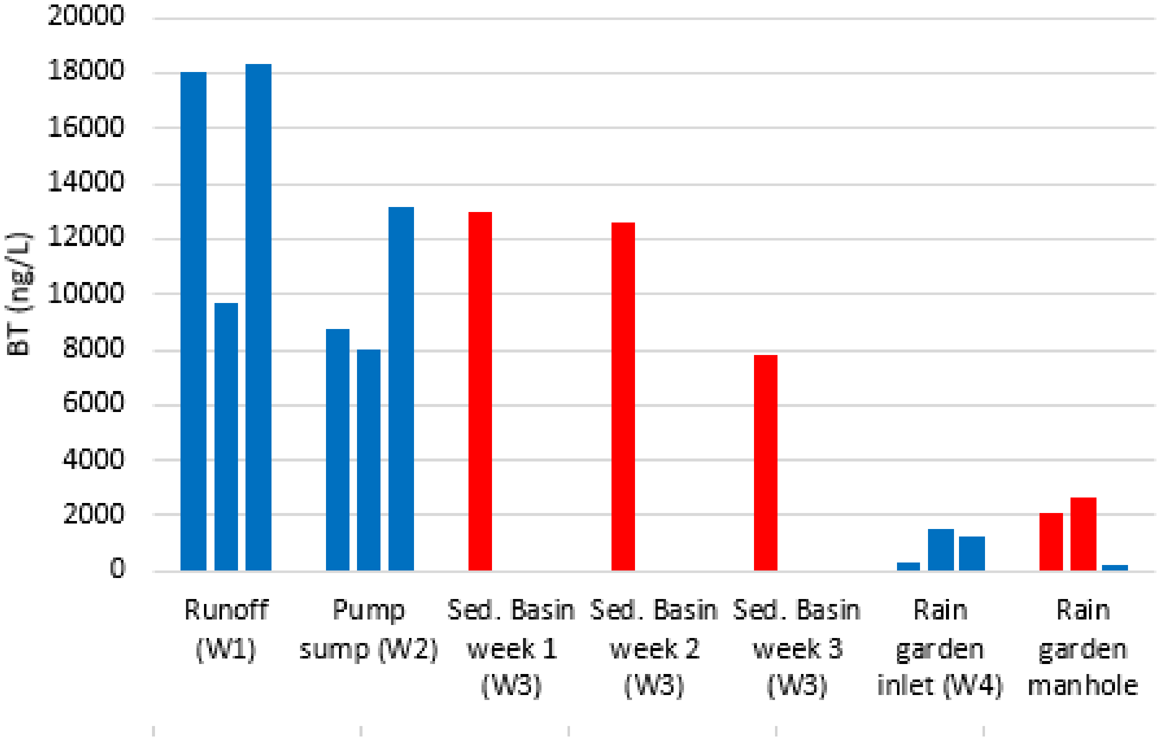

Figure 4.4: Concentration of Benzothiazole throughout the treatment system. Red colored bars have been stored one month at 4°C.

ABT

The concentrations of ABT in the wash water are low, and appears to be stable throughout the treatment system without any significant reduction in concentration (Figure 4.5). Even with low logKow of 2.00, it seems that ABT is settling, which may be explained by its low solubility of 310 mg/L. and occurrence in sediment samples (Table B.2). The third sample of runoff is quite low, but compared to other thiazole concentrations in tunnel wash water, the deviation of ABT is small. The peak in analyzed concentration at sedimentation basin week 1, 2 and 3 are probably, as with the other samples, explained by degradation in fridge. The time difference in analysis is not expressed in the rain garden manhole sample, which could imply that the time related degradation in fridge mainly occur in the samples early in the treatment system, and that the time related degradation are almost complete when collected in manhole. The peak concentration at sedimentation basin could also be caused by degradation of other benzothiazoles into ABT, thus there are no literature of such degradation to our far knowledge. The high concentrations in the rain garden inlet, are also expected due to dilution by untreated storm water. The mineralization of ABT is expected to be negligible, considering there is no chloride (road salt) present at this time of year.

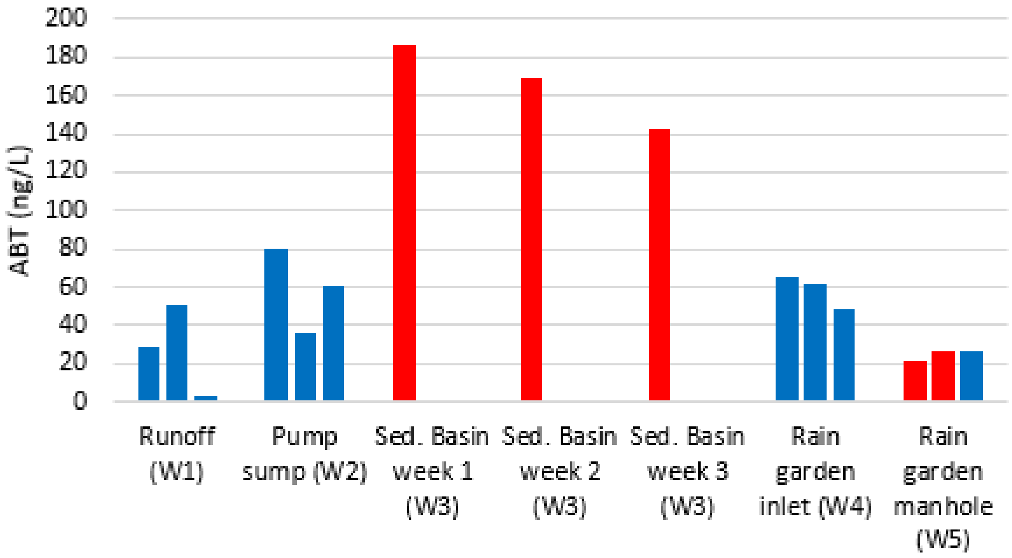

Figure 4.5: Concentration of ABT throughout the treatment system. Red colored bars have been stored one month at 4°C.

4.2.1 Sedimentation of the BTs

Without any inlet sampling as a reference point for the sedimentation process, the graph show 100% concentration after one week of sedimentation (Figure 4.6). This is not ideally to get an insight of the sedimentation paths of BTs over time. Nevertheless, the trend is decreasing as showed in 4.6. The removal of OHBT and MTBT is greater than ABT and BT. This can substantiate with the high logKow for both OHBT and MTBT.

In theory, the MTBT should adsorb more rapidly to suspended solid (SS) and sediment faster than OHBT, this may substantiate that OHBT undergo degradation during the sedimentation process, giving OHBT the greatest reduction. The reduction of MTBT is mainly due to sedimentation, thus MTBT is not expected to undergo any significant degradation considering its stable condition. The unexpected abrupt reduction of BT between the second week and the third, even with a high solubility, may indicate that there is a degradation of BT after >2 weeks. ABT has the lowest reduction, which can be expected due to its low logKow (low particle affinity). Brownlee et al. 1992 showed that logKow for MBT decreased with increasing pH. pH may affect the particle affinity of the other BTs as well and could be an explanation of the low settlement for BT and ABT (Subchapter 2.9.2) given the high pH in tunnel wash water(Aasum 2013).

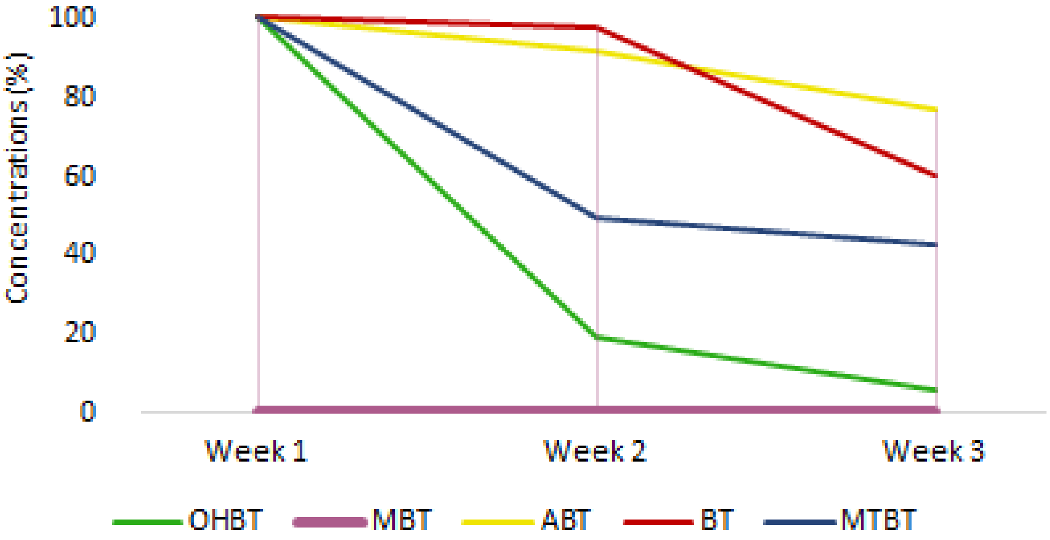

Figure 4.6: Distribution of BTs in sedimentation basin over 3 weeks effected by degradation and sedimentation.

4.2.2 Possible degradation of BT and MTBT

The three samples that were collected from the rain garden manhole, were analyzed in two rounds. Sample 1 and 2, were analyzed one month earlier than Sample 3, meaning that Sample 3 has been one month longer in the fridge. OHBT and ABT for all three samples are comparable and can indicate that these three samples are representative and that OHBT and ABT may not have undergone further degradation after they were collected in the field (Table 4.6).

Table 4.6: Three samples from rain garden manhole

	OHBT	**MBT**	**ABT**	**BT**	**MTBT**
Sample 1	179	<1	22	2131	1838
Sample 2	198	<1	27	2609	1819
Sample 3	201	<1	26	215	53

However there are drastic reductions of BT and MTBT after one month in the fridge (Sample 3). Reduction of BT and MTBT may have occurred due to transformation to other thiazoles than analyzed in this study, considering the stability of OHBT and ABT in these samples. MTBT were not expected to undergo any significant degradation due to its stability, according to literature (De Wever et al. 2001, Reemtsma et al. 1995) and to determine the stability of MTBT and BT as possible markers for TWP, further research is advised.

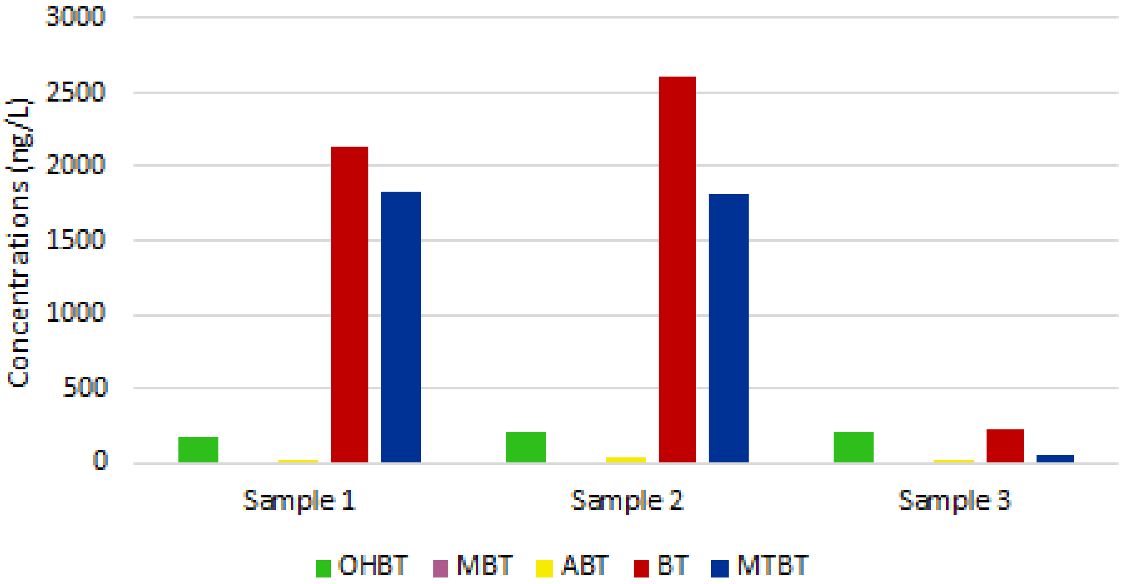

Figure 4.7: Three samples from rain garden manhole. Sample 3 is analyzed one month later than sample 1 & 2.

4.2.3 Infiltration efficiency of the BTs

The only samples comparable of studying the infiltration efficiency of the BTs in the raingarden, are the third sample from the rain garden inlet and the third sample from the rain garden manhole. This is because the date of analysis are equal (February 25^{th}), and the two samples are comparable since they are assumed from the same batch of water, before infiltration and after infiltration. The percentage reduction after infiltration is illustrated in Figure 4.8.

The sediment adsorption coefficient (logKow) was expected to have great influence on the restraining of BTS through the infiltration process, but the results were unexpected. MTBT has the lowest percentage reduction, even with its low affinity for water (Table 2.5). Furthermore did the low logKow values for both OHBT and BT seem to not affect the infiltation, when they had an reduction of approximately 80% each. This may indicate that there are characteristics of tunnel wash water, e.g. high pH, soap and oil, that possibly affects the logKow values and therefore the restrain in the rain garden. Other surroundings affecting the degradation of BTs can also be significant, like an expected higher bacteria content in the soil (biodegradation), compared to in tunnel wash water.

The high concentrations of OHBT and BT in the inlet sample can also indicate that treatment efficiency is greater for BTs present in high concentrations. This is substantiated by the low concentrations of MTBT and ABT in the inlet samples (Figures 4.3 and 4.5 respectively).

The presumable good treatment efficiency of BTs according to Figure 4.8 can be explained by the untreated stormwater affecting this step in the treatment process. The concentration of BTs in the inlet sample are presumably higher with the stormwater contamination, and can make the treatment efficiency appear better than it may have been without contamination of "fresh" BTs.

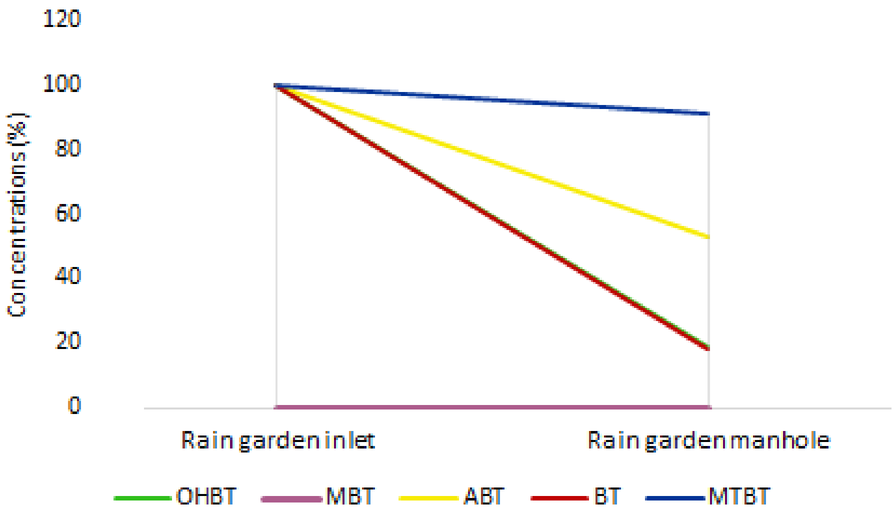

Figure 4.8: Treatment efficiency of BTs in the rain garden. OHBT is reduced at the same rate as BT.

4.3 Restraining and distribution of TWPs in the treatment system

The amount of TWPs restrained in the treatment system are difficult to determine considering leaching and degradation of benzothiazoles from TWPs. The rapid degradation in this study may be explained by the high pH of tunnel wash waters, which is known to affect the degradation of BTs (Brownlee et al. 1992) Since the measured BTs are leached from TWPs, and may have been transformed from other thiazoles or degraded over time, the measured concentration of BTs in the treatment processes gives no information about particle presence.

Lindholm 2015 stated that the gully pots could restrain smaller particles when <50% filling level in the grit chamber, and when the inlet water flow did not exceed 20−25 L/s. Even though gully pots are not considered a treatment process, according to Lindholm 2015 (gully pots 2.8), it may have been restrained TWPs in the gully pots during the tunnel wash. In Norway, the gully pots inside road tunnels are strictly contract−bound for emptying at certain filling levels before each tunnel wash. However, there are common practice of low maintenance of gully pots in open roads in Norway, meaning they may contain >50% filling level so their potential restraining capacity is reduced.

The TWPs are assumed to settle in the sedimentation basin when considering a retention time of three weeks, the specific gravity and average TWP grain size of 75 µm (Physical characteristics 2.6.1). The sedimentation process may be affected by soap and high pH (Brownlee et al. 1992). Assuming settlement of TWPs in sedimentation basins, there may be a minor quantity of TWPs entering the rain garden. In a normal situation, meaning that stormwater runoff is pumped directly to the rain garden, a larger TWP concentration may be transported to the rain garden.

Since the pump sump was not emptied accordingly to the washing plan, the sedimented wash water was "contaminated" by stormwater in the pump sump, meaning an addition of new TWPs. This "contamination" will primarily affect the first of the three rain garden inlet samples. The presence of TWPs in the first of the rain garden inlet samples are shown in the higher presence of OHBT which measured the same concentrations as the "pump sump" samples before sedimentation. The OHBT are expected to settle in sedimentation basin, as shown for sample 2 and 3 in Figure 4.2.

Based on Figure 4.8, there was a reduction of the dissolved BTs through the rain garden, indicating that the TWPs (of larger size) entering the infiltration system are most likely retained (Chapter 2.6.1).

4.4 BTs as tire wear tracers

Ideal criteria for tracers proposed by Wagner et al. 2018

- ***Be present in all tire materials in comparable portion, largely independent from manufacturer or manufacturing process.***
  In this study, one of the main focuses was detection of tire wear particles which are mainly generated from the tire treads. This will neglect the criteria for the BTs to be present in the whole tire structure like stated by Wagner et al. 2018. The concentration of BTs present in tire treads vary, according to literature used in this thesis. The differences in concentration may be due to human error or manufacturing. The latter should be assessed for further research.
- ***Not leach easily from tire particles into the surrounding environment.***
  The leaching itself is not a big concern when considering BTs as tracers. But knowing the right amount of leaching, on the other hand, is really important for further estimation of TP emission in the environment. According to literature, the leaching of BTs is affected by pH. This makes it important to assess leaching of BTs in various pH for any future studies on tire wear emission with BTs as tracers.
- ***Not be easily transformed while the tire particles reside in the environment (air, water, soil).***
  The extent of transformation (i.e. degradation) of BTs in this study are varying. The degradation processes are likely to be influenced by different factors e.g.

photodegradation (sunlight exposure), microbial degradation (exposure to bacteria), pH, and oxidation/mineralization. The photodegradation may not affect the results in this study, due to the cover of tunnel ceiling, but must be considered for the relevant BTs in further research in the environment. There are known microbial degradation paths for MBT and BT in active sludge systems with high concentration of bacteria. These concentrations are not expected to be present in the tunnel wash water in this study, but may affect the tracer suitability of some BTs. The rapid degradation of MBT and possibly OHBT, MTBT and BT, may be due to the alkaline water (pH 7−pH 10), which is stated by Brownlee et al. 1992. This high pH is not expected to be found in the environment, but should be taken into consideration to determine tracer suitability of BTs in road runoff where high pH may occur.

- ***Be sufficiently specific for tires, namely not present in relevant concentration in other traffic related particulate matter, such as brake dust, roadway particles or surface runoff.***

  As presented in Table 2.5, the presence of BTs in other traffic related sources are limited. Although, it is proven that some BTs (i.e. OHBT, MBT and BT) are present in antifreeze and that old tires are used in CRM-asphalt, which neither are expected to be present in this study. Considering this, tire treads are the only source related to BTs on Norwegian roads, except for antifreeze leaching from damaged vehicles.

- ***Have a concentration in tire material significantly higher than in the particles forming the sample matrix (soil, sediment, road dust, suspended matter, aerosol).***

  The particle of interest in this thesis are present in the environment as tire wear particles (TWPs), an aggregate particle consisting of "clean" tire tread particles (TPs) and minerals/dust from the carriageway. The concentration of BTs in a "clean" tire particle (TP) are much higher than in a TWP, and significantly higher than in the rest of the road dust matrix.

- ***Be analytically accessible by methods of high precision, accuracy, and sensitivity at reasonable analytical effort.***

  The benzothiazoles are detectable in scales of nanogram (ng), which is presumed analytically accessible.

5. Conclusion

Objective 1: Calculate tire wear emission in a tunnel based on the concentration of benzothiazoles in wash water runoff.

The concentration of BTs in the wash water runoff may give a good estimate of the total TP emission accumulated inside the tunnel, assuming limited transport of particles, degradation and minor exposure to water. Using the measured concentrations of MTBT, BT and the sum of BTs (ABT, MTBT, BT and OHBT) in wash water runoff resulted in estimated TP emissions comparable to earlier literature, used in equation 3.1, according to our calculations. This indicate that some of the selected BTs of this study may fit as tracer for TWPs in the road tunnel. The daily TP emission estimated from each of the BTs (Table 4.1), indicated a span from 0.1 to 10.4 kg TPs/day emitted in the Smestad tunnel. MBT are completely excluded as a tracer of TPs in this study, due to its rapid degradation in water, and unstable characteristics. ABT seems to give an estimated TP emission twice as high as by the other tracers. In other words is ABT deviating from the range 0.1−4.2 given by the other tracers. When compared to estimations given by Equation 3.1, the TP emission in the Smestad tunnel can possibly be expected in the range 0.1−4.2 kg TPs/day.

Objective 2: Assess the distribution of tire wear particles in a treatment system for tunnel wash water, based on concentrations of benzothiazoles.

In order to determine the TWP distribution in the tunnel wash water treatment system, the use of concentration of BTs as parameter were insufficient. In regards to leaching and degradation of BTs from TWPs in the water phase, the concentrations of BTs throughout the treatment system were not a valid parameter to determine retention of TWPs. The treatment system is constructed for restraining TSS, meaning the dissolved BTs are passing through the process and can not confirm actual particle presence at any step in the treatment process. It is reason to believe that the basic wash water, combined with soap, is a large contributor to the rapid degradation of BTs in this study. Concentration of BTs in the treatment steps of tunnel wash water are therefore unsuitable for determining of the presence of TWPs throughout the treatment system.

For future research, by measuring BTs in dry soil, where exposure of rain and sunlight is minor, it may be possible to reduce the uncertainties regarding degradation and the calculation of TP emission may be more valid. Road tunnels can possibly be good sampling sites for measuring BTs in road dust.

It may be a possibility using concentrations of all thiazoles to reduce the uncertainties considering degradation. By testing for all BTs, the sum of thiazoles could possibly give a better estimate when determining TWPs presence in the environment.

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Vedlegg A: Liquid sample results from laboratory

Table A.1: Mixed samples of wash water runoff from 3 different locations in the tunnel. Liquid samples with concentration in ng/L.

	OHBT	**MBT**	**ABT**	**BT**	**MTBT**	**Sum**
Sample 1	1 305	<1	29	18 059	211	19 604
Sample 2	1 530	<1	51	9 651	312	11 544
Sample 3	1 445	<1	3.5	18 319	148	19 915.5

Analyzed 25 February 2019

Table A.2: Mixed samples from the pump sump at three separate times; early, middle and late in the filling process. Liquid samples with concentration in ng/L.

	OHBT	**MBT**	**ABT**	**BT**	**MTBT**	**Sum**
Sample 1	1 375	<1	80	8767	132	10 354
Sample 2	1 655	<1	36	7 970	212	9 882
Sample 3	1 895	<1	61	13 185	234	15 375

Analyzed 25 February 2019

Table A.3: Mixed samples from the sedimentation basin after 1, 2 and 3 weeks of sedimentation, respectively. Liquid samples with concentration in ng/L

	OHBT	**MBT**	**ABT**	**BT**	**MTBT**	**Sum**
Sample 1	301	<1	186	12 966	5 247	18 700
Sample 2	56	<1	169	12 589	2 578	15 392
Sample 3	16	<1	142	7 776	2 220	10 154

Analyzed 31 January 2019

Table A.4: Mixed samples from the rain garden inlet at three separate times: early, middle and late in the emptying process of the sedimentation basin. Liquid samples with concentration in ng/L

	OHBT	**MBT**	**ABT**	**BT**	**MTBT**	**Sum**
Sample 1	1 685	<1	66	315	83	2 149
Sample 2	1 030	<1	62	1 496	141	2 588
Sample 3	1 050	<1	49	1 208	58	2 365

Analyzed 25 February 2019

Table A.5: Mixed samples from the rain garden manhole at three separate times: early, middle and late of the rain garden infiltration process. Liquid samples with concentration in ng/L

OHBT	MBT	ABT	BT	MTBT	Sum	
Sample 1 *	179	<1	22	2 131	1 838	4 170
Sample 2 *	198	<1	27	2 609	1 819	4 653
Sample 3	201	<1	26	215	53	495

*Analyzed 31 January 2019

Analyzed 25 February 2019

Vedlegg B: Sediment sample results from laboratory

Table B.2: Mixed sample of sedimented sludge from sedimentation basin after emptying. Liquid sample with concentration in ng/L.

	OHBT	MBT	ABT	BT	MTBT	Sum
Sedimented sludge sample	4 660	<1	78	2 293	143	7174

Analyzed 12 February 2019

Table B.3: Mixed sample of sediments from sedimentation basin after emptying. Sediment sample with concentration in ng/g.

	OHBT	MBT	ABT	BT	MTBT	Sum
Sediment sample	141	56	2.3	32	17	248.3

Analyzed 12 February

Table B.1: Sediment samples from three different gully pots in the tunnel. Concentrations in ng/g sediment.

	OHBT	MBT	ABT	BT	MTBT	Sum
Sample 1	161	16	1.9	23	10	211.9
Sample 2	301	17	1.6	42	20	381.6
Sample 3	181	32	1.8	33	38	285.8

Analyzed 25 February 2019

Table B.4: Mixed sample from the rain garden top soil before and after infiltration. Sediment samples with concentrations in ng/g.

	OHBT	**MBT**	**ABT**	**BT**	**MTBT**	**Sum**
Before infiltration	2.6	0.9	0.7	<10	7.2	11.4
After infriltration	27	0.7	1.7	<10	15	44.4

Analyzed 12 February

Vedlegg C: Drawing of the Smestad tunnel

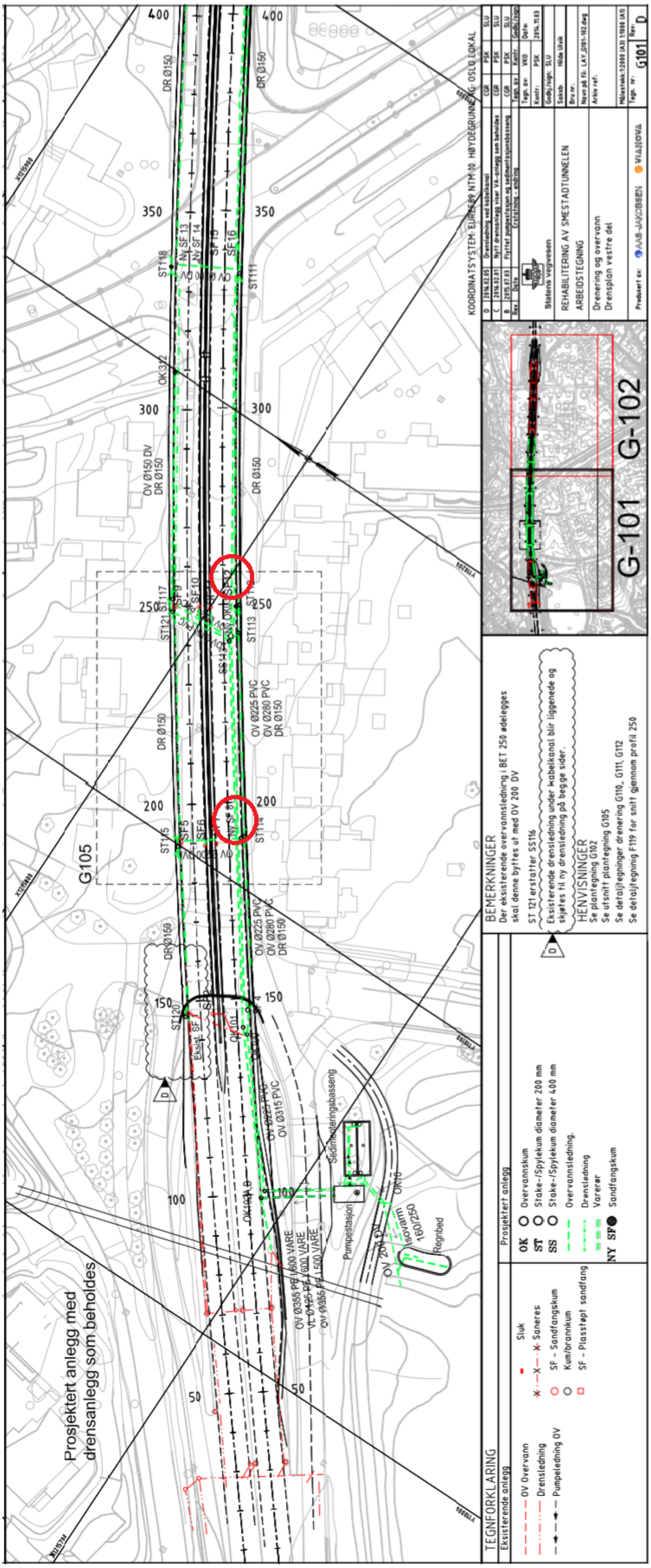


Figure C.1: The smestad tunnel with markings on two of the three gully pots sampling locations

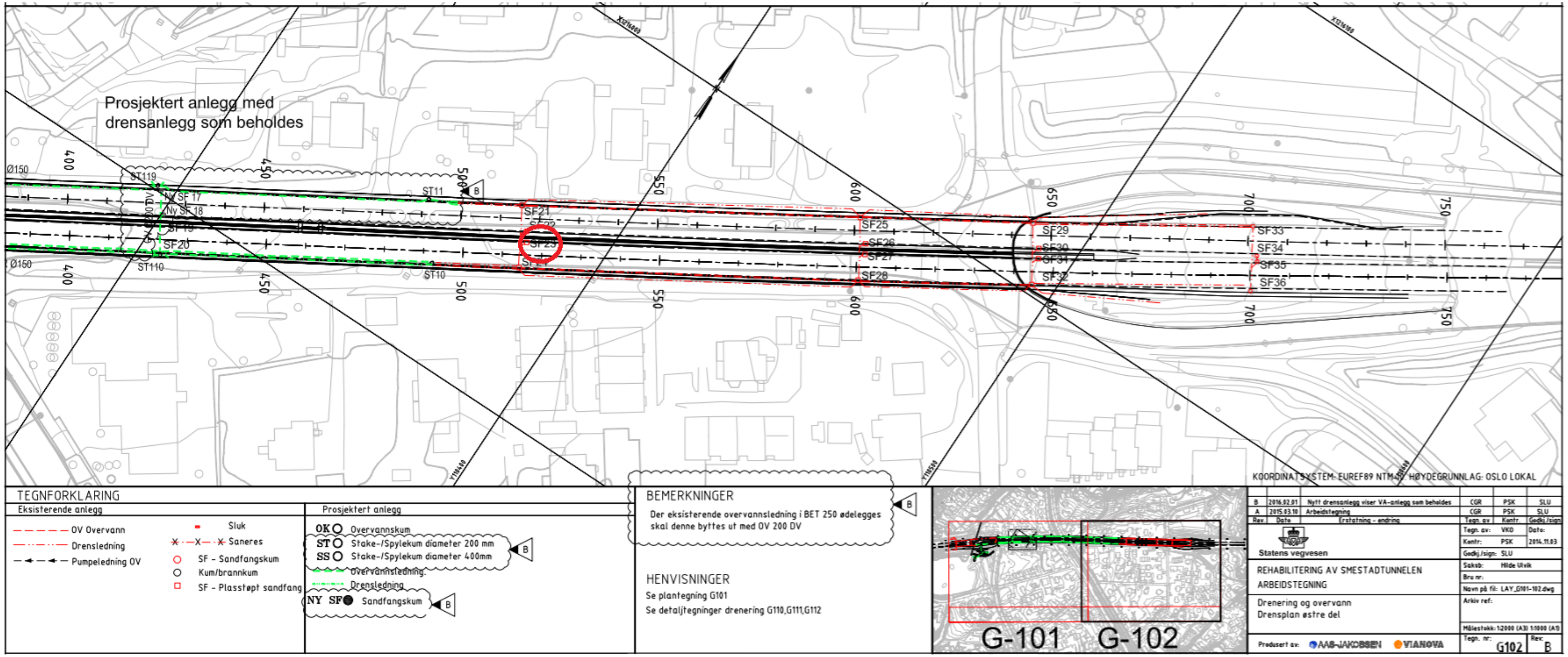


Figure C.2: The smestad tunnel with markings on one of the three gully pots sampling locations

Vedlegg D: Analysis of BTs by NIVA

Analysis of Benzothiazoles with UPLC–MS

Sample extraction and clean up

Sediment samples: About five g of homogenized sample (wet sediment) was weighed into a 50 mL tube and prior to extraction 20 µL of internal standard (1 ng/µL of d4-imidacloprid) was spiked into the sample. A volume of 8 mL of methanol (MeOH) and 100 µl formic acid (cons) was added into the tube. The sample tube was mixed by vortex for 1 min followed with sonification for 30 min at room temperature. The extraction process was repeated with another 4 mL of MeOH and formic acid and the extracts were combined.

Water samples: About 400 mL of water spiked with 20 µL of internal standard (1 ng/µL of d4-imidacloprid) was loaded on a MeOH activated 500 mg Oasis HLB (Waters) solid phase column. The BTs were eluted of the column with 6 mL of MeOH.

All samples were filtered with 20 µm Spin-X centrifuge nylon filter (Corning) and finally transferred into a LC vial ready for UPCL-MS analysis.

UPLC-MS analysis

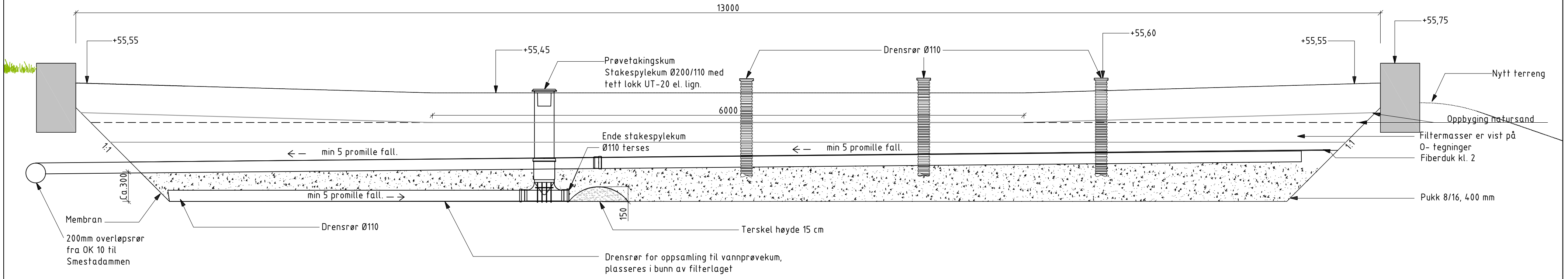
Liquid chromatography was performed on an Acquity HSS C18 column (1.8 µm, 100 × 2.1 mm) (Waters, Milford, MA, USA), using a Waters Acquity UPLC module. Separation was achieved using linear gradient elution at 0.5 mL/min starting with acetonitrile (MeCN)–water (5:95, water containing 0.1% formic acid and 5.2 mm ammonium acetate for positive and negative ionization respectively) rising to 100% MeCN over 9 min. Isocratic elution with 100 MeCN was maintained for 2 min before the eluent was switched back to 5% MeCN. The UPLC system was coupled to a Quattro Premier XE tandem mass spectrometer operating with an ESI interface (Waters Micromass, Manchester, UK). Typical ESI parameters were a spray voltage of 3.5 kV, desolvation temperature at 400 °C, source temperature at 120 °C and cone gas and desolvation gas at 50 and 800 L/h of N2, respectively. The mass spectrometer was operated in MS/MS mode with argon as collision cell gas at 1.5×10^{-3} Torr. Ionization and MS/MS collision energy settings (typically 25 eV) were optimized while continuously infusing (syringe pump) 20 ng/mL of individual BTs, at a flow rate of 5 µL/min. Screening of BTs were performed with multiple reaction monitoring (MRM) in positive ionization mode; ABT 151>108.8, BT 136>108.8, MeBT 150>108.8, MTBT 182>167 and in negative ionization mode; OHBT 150>134 and MBT 166>134.

Vedlegg E: Rain garden and sedimentation basin

Detail drawings by Aas- Jakobsen and Vianova, of the rain garden and the sedimentation basin connected to the Smestad tunnel.

Regnbed

Lengdesnitt midt av regnbed



Regnbed

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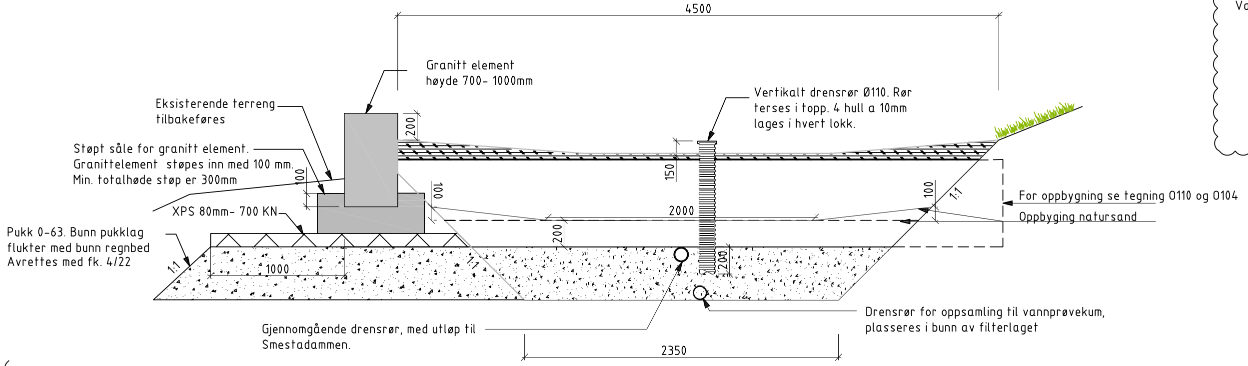
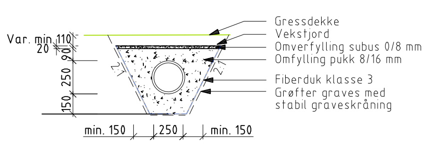


Snitt A - A

Snitt isovarmrør fra regnbed til OK 10



Snitt B - B

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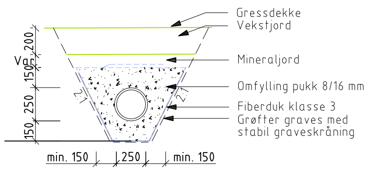

Regnbed

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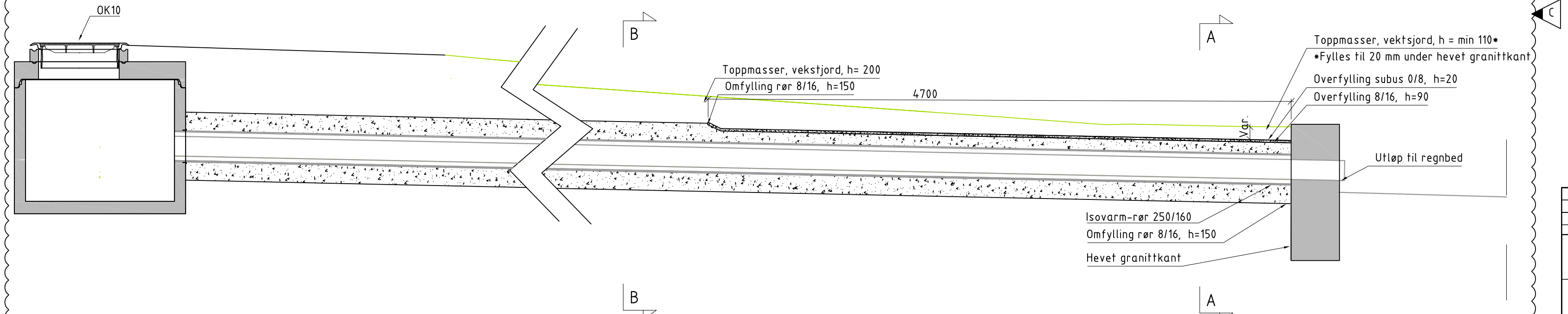


Henvisning:
G 101 /G103 -Plantegning drensanlegg
O 103,104,110 Detaljtegning regnbed
KOORDINATSYSTEM: EUREF89 NTM 10. HØYDEGRUNNLAG: OSLO LOKAL

Rev.	Dato	Erstatning - endring	Tegn. av	Kontr.	Godkj./sign.
C	2016.04.05	Oppbygging masser for isovarm-rør	CGR	PSK	SLU
B	2015.10.28	Fundament kantstein, oppbygning natursand	CGR	PSK	SLU
A	2015.03.10	Arbeidstegning	CGR	PSK	SLU

Statens vegvesen

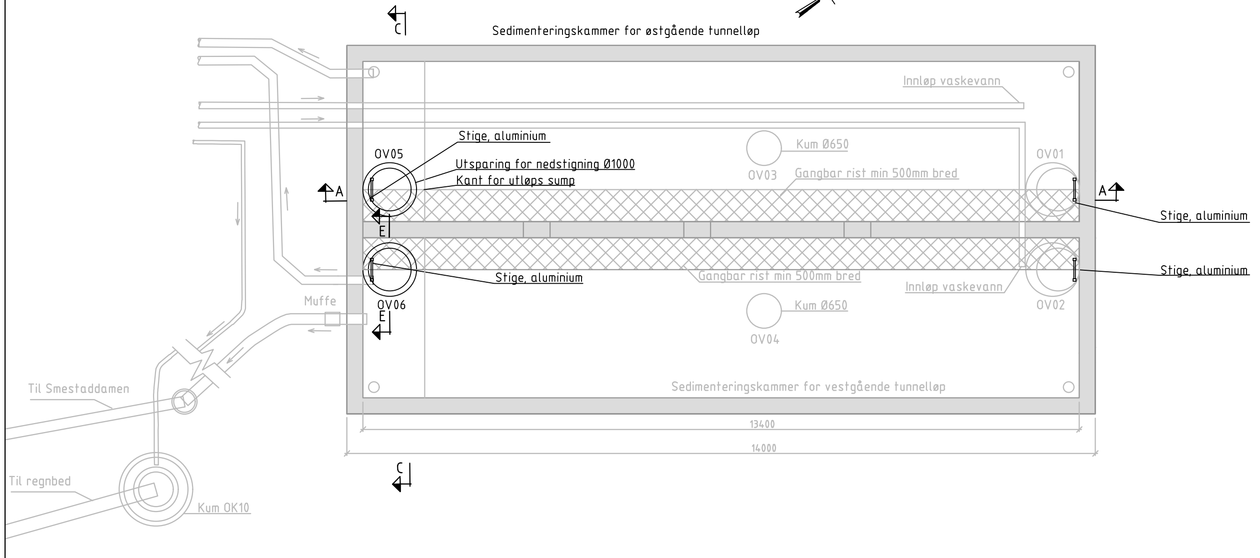
REHABILITERING AV SMESTADTUNNELEN
ARBEIDSTEGNING

Drensanlegg, detaljer.
Drensledning og stakekummer i regnbed

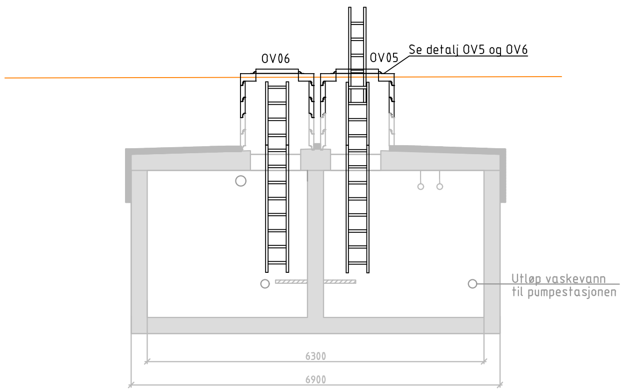
Tegn. av: CGR
Kontr: PSK
Dato: 2014.11.03
Godkj./sign: SLU
Saksb: Hilde Ulvik
Bru nr:
Navn på fil: LAY_G_Detaljer.dwg
Arkiv ref:
Målestokk:1:40 (A3) 1:20 (A1)
Tegn. nr: G116
Rev: C

Produsert av: AAS-JAKOBSEN VIANOVA

Sedimenteringsbasseng
Plan



Sedimenteringsbasseng
Snitt C-C



Detalj OV05 og OV06
Plan
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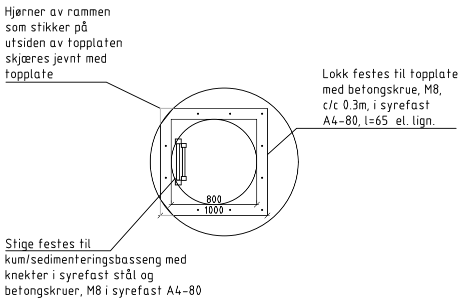
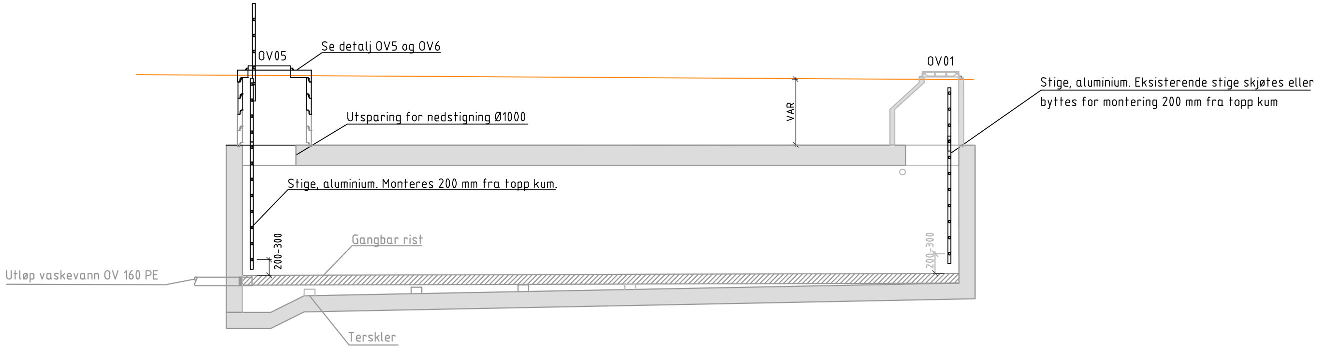
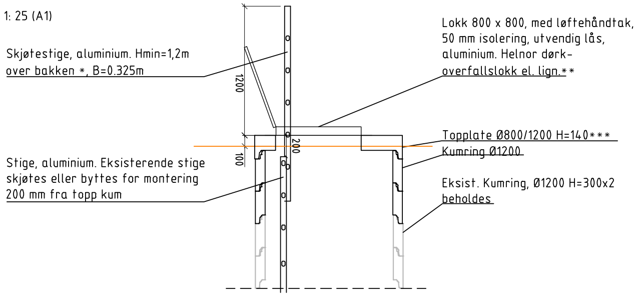


Sedimenteringsbasseng
Snitt A-A



Detalj OV05 og OV06
Snitt E - E
M: 1:50 (A3), 1: 25 (A1)



*	Kun en skjøtestige
**	Lokkets hengsler er på lik side som stigen monteres.
***	Topplate stikker 100 mm over terreng for enklere tilkomst og lokalisering av nedstigningskum

KOORDINATSYSTEM: EUREF89 NTM 10. HØYDEGRUNNLAG: OSLO LOKAL

Rev.	Dato	Erstatning – endring	Tegn. av	Kontr.	Godkj./sign.

Statens vegvesen

Tegn. av: CGR	Dato:
Kontr: PSK	2014.11.03
Godkj./sign: SLU	
Saksb: Hilde Ulvik	
Bru nr:	
Navn på fil: LAY_G300.dwg	
Arkiv ref:	
Målestokk: 1:100 (A3) 1:50 (A1)	
Tegn. nr: G300	Rev:

REHABILITERING AV SMESTADTUNNELEN
ARBEIDSTEGNING

Smestadtunnelen, sedimenteringsbasseng
Kumtopp og stiger

Produsert av: AAS-JAKOBSEN VIANOVA

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Noregs miljø- og biovitskapelege universitet
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